

# Corrosion

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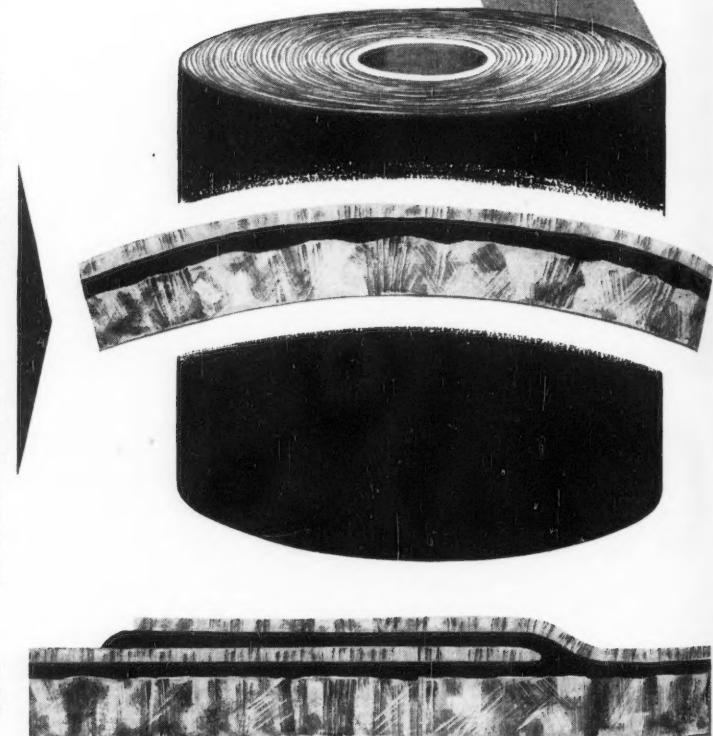
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# This Month in Corrosion Control . . .

SOME REASONS for corrosion control connected with the necessity of conserving exhaustible mineral resources are considered in this month's editorial on Page 6.

MULTIPLE COOLING TOWERS can be treated with inhibitor by means of an ingenious central feed system described in an article beginning on Page 9.

EFFECT OF VARIABLES, including salinity, oxygen saturation and seasonal freshening affect a cathodic protection system on steel piling in an estuary. Some of the details of a system protecting steel piling are covered in an article starting on Page 14.

WHAT HAPPENED at the Dallas NACE 16th Annual Conference and Corrosion Show is described and illustrated in some detail in a report on the conference beginning on Page 41. As usual, the 5-day meeting was overflowing with events, with an adequate icing of social affairs.

IMPORTANCE OF PLASTICS to the military is underlined by the establishment at Picatinny Arsenal of a Plastics Technical Evaluation Center. Some information on this new Department of Defense activity is given on Page 65.

BOOKS ON CORROSION are being printed in greater numbers than ever. Seven items in the monthly "Book News" column will be of interest to those who are building a corrosion control library. Turn to Page 70.

WHILE EXTREMELY STABLE when pure, stabilized and isolated from its environment, hydrogen peroxide reacts violently with certain materials. Because this chemical is being used with increasing frequency in a wide variety of industrial reactions, data on materials of construction for use with hydrogen peroxide beginning on Page 100 should be interesting to many.

COMPLEX INTERRELATED EFFECTS of aluminum and iron content, current density and time on the performance of zinc galvanic anodes in sea water

are considered in detail in an article beginning on Page 107. A novel 3-variable presentation of graphed data is included.

EFFECTIVE DEOXYGENATION of boiler water can be effected by diffusing the oxygen into a gas mixed with the water. The gas absorbs the oxygen. The gas and oxygen are removed from the water and the oxygen scavenged with charcoal. See Page 114 for complete information.

ALUMINUM CORROSION PRODUCTS are analyzed in detail in a report of NACE Technical Unit Committee T-3-B on Corrosion Products. This report, beginning on Page 117, considers corrosion products formed in fresh and salt water and the atmosphere.

MORE DEFINITIVE DATA ON ZIRCONIUM and its alloys are given in the article starting on Page 124 concerning the effect of carbide inclusions on oxide film failure of this metal used extensively in atomic piles.

OXIDATION BEHAVIOR of the refractory metals, whose growing applications in atomic energy and in the chemical industry make them especially significant, is explored in a thorough, fully referenced article beginning on Page 129.

MORE DATA ON HYDROGEN, this time on its absorption from water and aqueous solutions, are given in this issue. The article is a partial sequel to a paper by the same author published in December, 1958. See Page 134.

WHAT HAPPENS TO AN OILWELL INHIBITOR at high temperatures? This important problem is explored in detail by a working oil company researcher, who gives tabulated results of average protection as measured by weight loss. For the full story see Page 137.

HOW CORROSION ATTACKS WELDED low alloy steel is surveyed by three Canadian researchers in an article beginning on Page 141. This article is a sequel to one published in January, 1959.





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Boiler Model Tests of Materials for Steam Generators in Pressurized Water Reactor Plants, by E. Howells, T. A. McNary and D. E. White.

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Cathodic Protection of Hulls and Related Parts, Technical Committee Report T-3G-1.  
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## How to Postpone the Days of Balance

**T**HREE ARE TWO DAYS OF BALANCE facing the United States with respect to its production of ferrous metals. The first, and most important of these will come when loss of iron to corrosion balances the total output of ferrous metals. The second, probably even more imminent and threatening in some aspects, is the day when Soviet Russia's steel output equals ours.

There is no known accurate estimate of the tonnage of iron now in use in the United States. There is no estimate of any stature on the loss to rust of iron in use. The United States produced 2,620,000,000 tons of steel in 40 years.\* If half of this amount is still in use today, or 1,310,000,000 tons, corrosion losses would have to be 7.6 percent a year to equal 100,000,000 tons, nominal current production of U. S. mills.

Unfortunately no one knows at what rate steel in use is corroding. The losses may be at a much greater rate than 7.6 percent, or at a much lower rate, and the tonnage in use may be (and probably is) considerably greater than 1.31 billion tons.

The first day of balance, if it ever is reached, will mean to some extent, that our gain in ferrous capital goods has come to a halt. This is modified by other unknown factors such as the rate at which non-ferrous metals take over the role of steel; improvements in design and strength which permit smaller tonnages to do more work; shifts from metal to non-metal construction and last but not least, the success of corrosion control efforts on all exposed metals.

The second day of balance, while potentially nearer, may never be reached, for obvious reasons. Among the reasons Soviet Russia's production of steel may never equal that of the United States is the fact that by natural laws a rate of gain tends to diminish

as quantities increase. The second is that even modest rate increases in United States' output represent a much greater tonnage than increases at a like rate against the Soviet base of production.

Another factor which needs to be weighed is the efficiency with which the produced material is used. If U. S. efficiency can be kept ahead of the Soviet's, the tonnage gains assume far less importance.

Furthermore, if this nation is able to slow down corrosion losses of iron appreciably, the savings from this source alone may equal Soviet Russia's entire output. This serves to underline the need for greater emphasis on corrosion control for all metals.

As the world gets closer to exhaustion of its metals, it becomes increasingly important to preserve available metals in use. This means some thought should be given to abandoning present practice of permitting metals to disintegrate and disperse into forms from which they cannot be recovered. It contemplates a change in the current thinking in the United States which results in the arbitrary discard of many components which may have failed only in one small area. It presents a challenge to industry and perhaps to government to reassess our system in which it often costs less to replace a unit than to repair it.

Conservation needs to become as common a word in discussing metals as it has in considering hydrocarbon fuels, wildlife, soil, water and wood. We may discover, while changing our point of view on our exhaustible metal resources, that we will find so many more efficient and desirable ways to do things that the whole effort may pay its own way.

\* The Battle for Steel Supremacy. By Alexander Gakner. Iron and Steel Engineer, Vol. 36, No. 11, 117-124 (1959) Nov.



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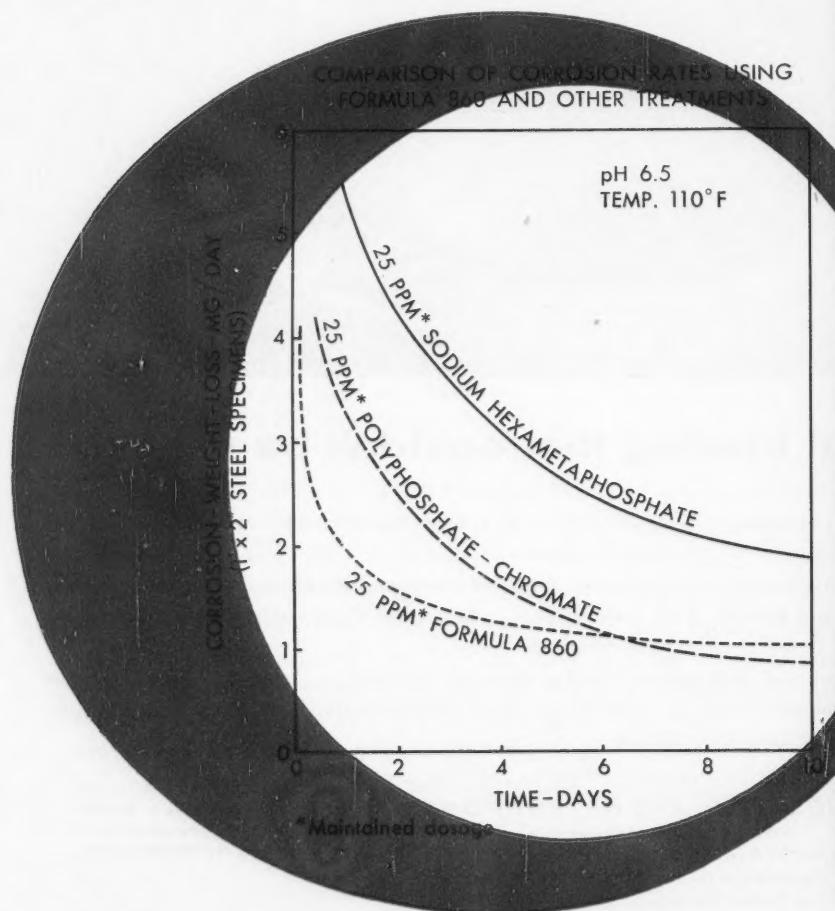
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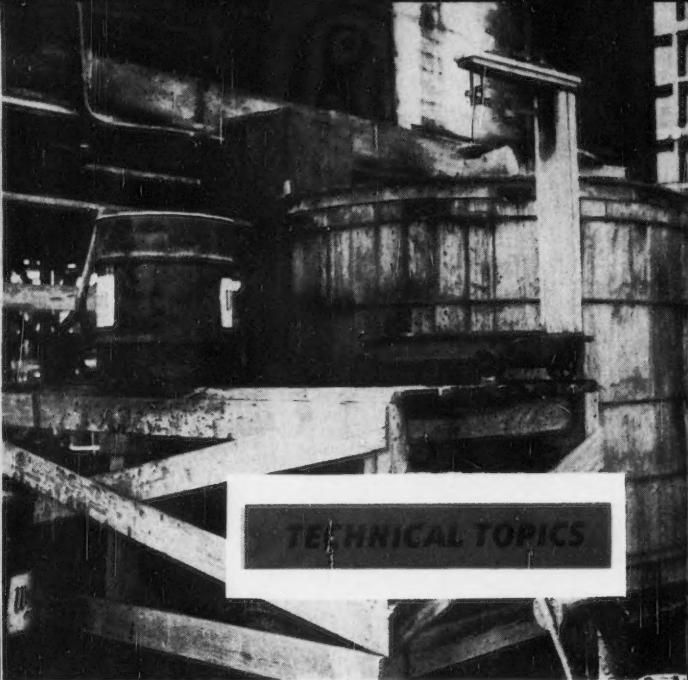
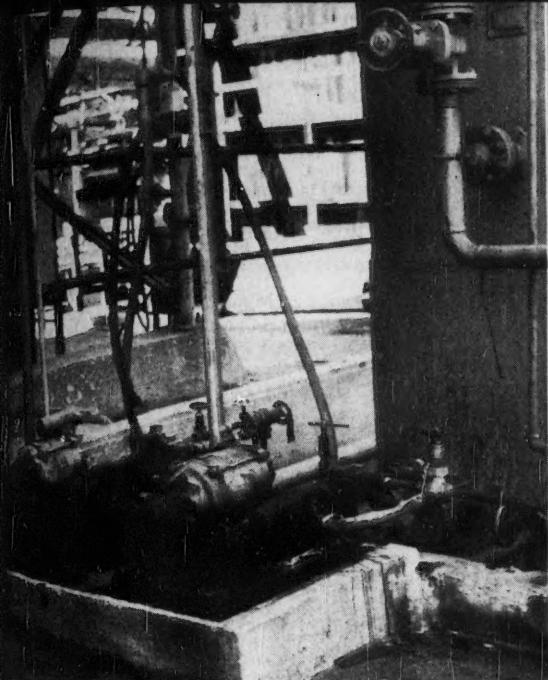
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TECHNICAL TOPICS

**CHEMICAL AND ACID PUMPS** (on the left) and corrosion inhibitor mixing tank (on right) are used in a centralized chemical feeding system for multiple cooling towers. A fluorocarbon-base paint was used to help prolong the life of the acid pumps. Corrosion had caused replacement of the pumps' exterior portions, resulting from acid drippage. The 1800-gallon mixing tank shown above is charged weekly. The inhibitor mixture is fed continuously at a constant rate through an adjustable pump.

#### Abstract

Describes feeding corrosion inhibitor and acid for pH control to several cooling towers from a single station. Problems encountered and their solutions are given. This system has provided excellent corrosion control for eight years. Advantages include low labor cost which averages one man-hour per day for entire cooling system. 7.4.1

**R**ECONIZING THE fact that good chemical control is necessary to obtain best results from water treatment, Tidewater Oil Company explored the feasibility of developing a centralized chemical feed in the multiple cooling tower system at its natural gasoline plant.

The plant is on a 1000-foot mountain top at the head of a valley in the mountains immediately inland from Ventura, Cal. Raw water supplied by the city of Ventura is chlorinated water drawn from wells.

#### Chemical Feed Problem

In most plants, chemical feed and control is applied to single cooling systems; thus, chemicals are added to each system, and each system is controlled as a single unit. Though practical in many cases, this was considered too expensive for the cooling systems in the Ventura plant.

The five Ventura cooling systems use an aggregate 155,000 gallons of water per day. Cost of manual additions of acid and inhibitor at each tower would be relatively expensive. Capital outlay for the installation of automatic acid feed at each tower would be several thousand dollars each, and total cost would be disproportionate to relative use.

\*Revision of a paper by William Harrison, Tidewater Oil Co., Ventura, Cal., and P. G. Bird, Wright Chemical Corp., Chicago, Ill., presented at the Western Region Conference, National Association of Corrosion Engineers, Bakersfield, Cal., September 29-October 1, 1959.

## Centralized Chemical Feed to Multiple Cooling Towers\*

#### Two Solutions Considered

Two solutions for developing centralized chemical feed were considered. One was to install a single tower with sufficient cooling capacity for the entire plant. This was considered at the time the old atmospheric towers were replaced by individual induced draft towers, but the plant layout and cooling requirements ruled out this possibility. The second and only practical solution was a centralized feed system.

Several methods of accomplishing central chemical feed were investigated. Addition of acid posed the greatest problem. Piping acid directly to each tower from a central point did not seem to be feasible. The only alternative was to feed acid to the water line supplying the towers. Because the make-up water has a high bicarbonate content, the water would be corrosive after acid addition due to its free CO<sub>2</sub>. Aeration to remove

(Continued on Page 10)

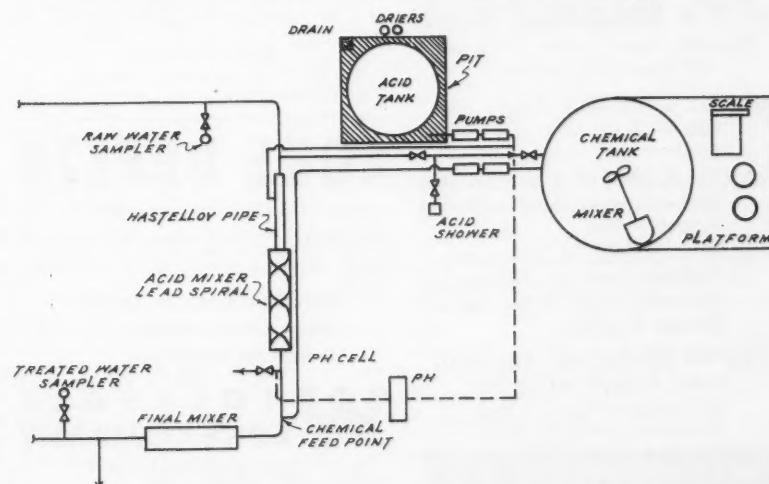


Figure 1—Diagram showing chemical and acid feed system that treats all water fed into the multiple cooling tower system.

## Cooling Towers—

(Continued From Page 9)

the carbonic acid would result in less corrosive water in the supply line, but suitable equipment would be costly and entail repumping. The acid feed problem thus resolved itself into what to do about the make-up water feed line.

Acid resistant piping could be installed, but, since good piping was in place, the suggestion was made to pump the acid and corrosion inhibitor into the existing steel make-up line and to consider the line expendable. Initially, this suggestion was thought to have dubious merit, but because only the supply line would be affected, it was given further consideration. One reason was that because the excess CO<sub>2</sub> would be released from the water during the first pass through the towers, CO<sub>2</sub> in the make-up water would not have appreciable effect on the pH of the circulating water. Computations indicated that the amount of fresh water going to the heat exchange equipment is only 2 to 3 percent of the recirculation, depending on the amount of evaporation. A 20 F temperature drop across the tower corresponds to a 2 percent evaporation loss. This, with windage and blowdown, amounts to a bit more than 2 percent of the recirculated water to be made up of fresh water. Finally, the supply line was located above ground and was readily accessible for repair.

It was anticipated that the make-up line would require replacement at intervals from three to five years, and this expense could be absorbed as an operating cost rather than carried as capital outlay. Eight years later, the line has not required replacement and recent inspection disclosed that there were sev-

eral more years of life to be expected from the line.

The line consists of four-inch uncoated steel pipe. This line has been carrying water at pH of 4.5 to 5.0 and occasionally lower, with only 8 to 10 ppm of corrosion inhibitor present.

Amount of corrosion inhibitor present in the feed line is below its effective range because it is concentrated five to six times in the towers. Thus, the amount of inhibitor in the feed line is only  $\frac{1}{6}$  to  $\frac{1}{5}$  that in the cooling water.

Water requirements for each tower are controlled by float valves. These are polychloroprene-lined butterfly-type valves. The pH recorder-controller, chemical and acid tanks, and associated equipment are near Tower 6.

Some difficulty was encountered with the automatic pH controller, resulting in the addition of too much or too little acid. These variations have not caused appreciable damage to the feed line, tower or equipment because the acid feed was corrected promptly.

### Chemical-Acid Systems

Figure 1 shows the chemical and acid feed systems. Raw water passes through a high alloy pipe where the acid is added, then to a mixing chamber after which the corrosion inhibitor is added. Water then goes to final mixing chamber. There is a pH sampler just forward of the point where inhibitor is added.

The 2700-gallon acid storage tank is connected to a positive displacement acid pump equipped with a d-c variable speed motor controlled by the pH recorder-controller.

The pH response to acid addition is rapid, and no cycling has been experienced. The recorder-controller has a analog circuit, and the time lag between the point of acid addition and the pH electrode is short.

Greatest difficulty in the acid feed system has been with the pump. Corrosion caused the replacement of exterior portions of the pump because of acid drip-page. A fluorocarbon-base paint has prolonged the life of these parts; previous repairs had been required every 3 or 4 months. A lead trough was hung under the pump shaft to carry off acid that escaped through the seal.

The corrosion inhibitor is fed directly from the chemical vat which holds 1800 gallons and is charged weekly. The inhibitor is fed continuously at a constant rate through an adjustable pump.

### Water Sampling

A raw water sampling station is on the line immediately forward of the high

alloy pipe. Finished water is sampled from the line leading to cooling Towers 5, 6 and 11.

These samplers are operated by a timer that opens a solenoid valve every 5 minutes. The water flows through the orifice and shut-off valve into a five-gallon glass bottle so that daily composites are obtained.

### Mixing Chambers

In the acid mixing chamber, the acid rises into the four-inch high alloy pipe through a six-inch length of high alloy tubing; back-flow is controlled by check valve. The steel acid feed line originally was connected directly to the four-inch high alloy pipe but lasted only a short time. After a six-inch acid resistant nipple was installed on the line, no difficulty was experienced. The acid mixing chamber is lead lined; a lead spiral extends its length.

Acidified water is delivered to the pH cell through a half-inch steel line fitted with an orifice that controls a small flow through the cell, which in turn discharges to the basin of Tower 6.

Inhibitor is pumped into the line downstream from the pH sampling point to prevent buffering action of the inhibitor. It flows into the line from above through a steel nipple. Some mixing of acidified water and inhibitor takes place at an elbow, and complete mixing takes place in the final mixing chamber. This chamber is a straight 8-inch diameter steel tube, similar to the acid mixing chamber but is not lead lined and does not contain a spiral mixer. The pH of the water from the final mixer is substantially that of the water going to the pH cell because the inhibitor has negligible influence on pH.

There are three requirements for successful operation of a centralized feed system. The first is to obtain reliable feeding equipment for the acid and inhibitor; secondly, good control of blowdown from each tower; and third, systematic chemical testing of recirculating water in each tower. The latter is necessary for adjustments in blowdown and correction in chemical feed, and also serves as a check on the automatic pH controller.

### Training Personnel

The key to maintaining good chemical testing and control lies in securing cooperation of supervisors and operating personnel and their understanding the importance of water control. Supervisors were trained in the laboratory before becoming supervisors. As a result of this training almost no difficulty has been experienced stemming from poor control over week-ends and holidays.

The most critical part of the water treatment is acid addition. Because the pH recorder is easily observed, day or night, operators have ample opportunity to check it on their rounds. Supervisory personnel know how to check the recorder-controller to determine if it is in working order. Because of proper training the entire operation has been extremely satisfactory.

The testing procedure initially was rather elaborate but is now a simple routine. The total weekly time for laboratory testing and maintenance is:

- 2 hours collecting water samples ( $\frac{1}{2}$  hr. per day for 4 days)
- 2 hours laboratory chemical tests
- 2 hours clean electrode and pumps; inspect blowdown orifices and clean if necessary

(Continued on Page 12)

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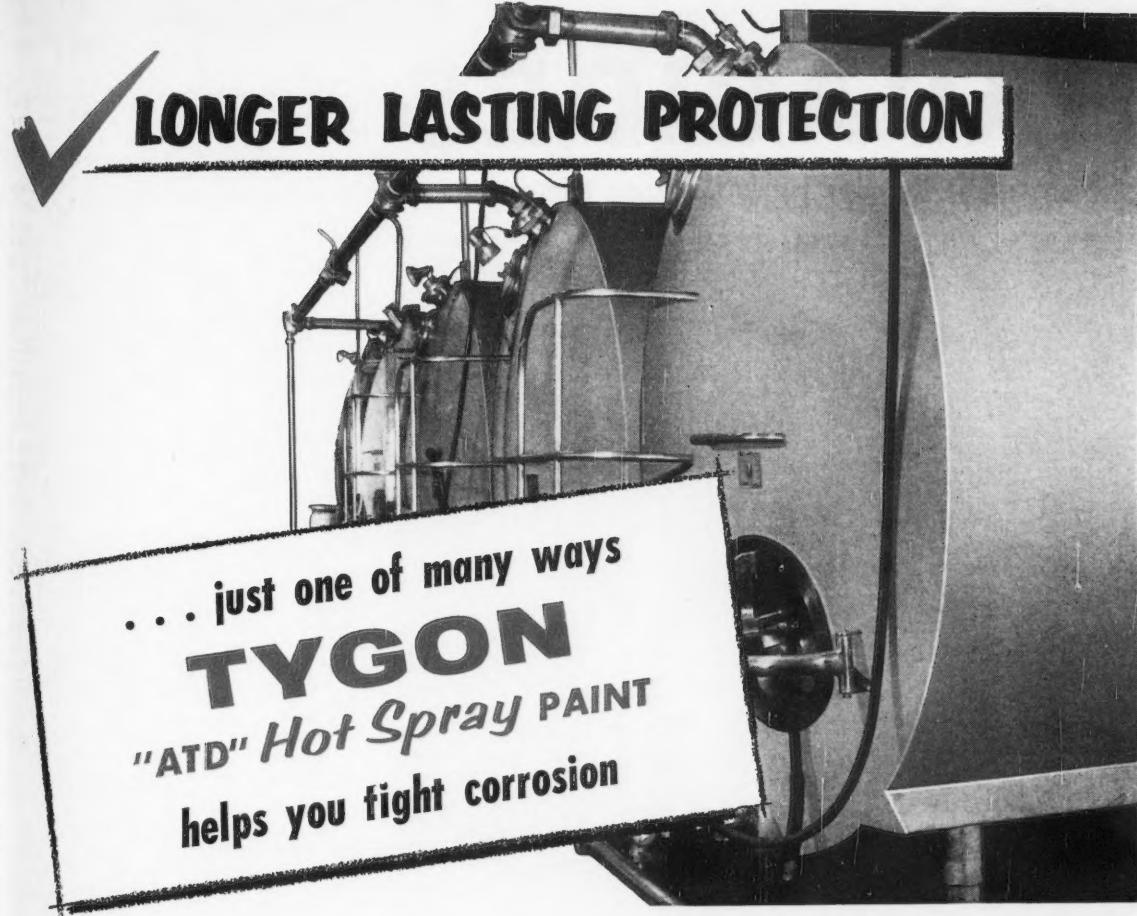
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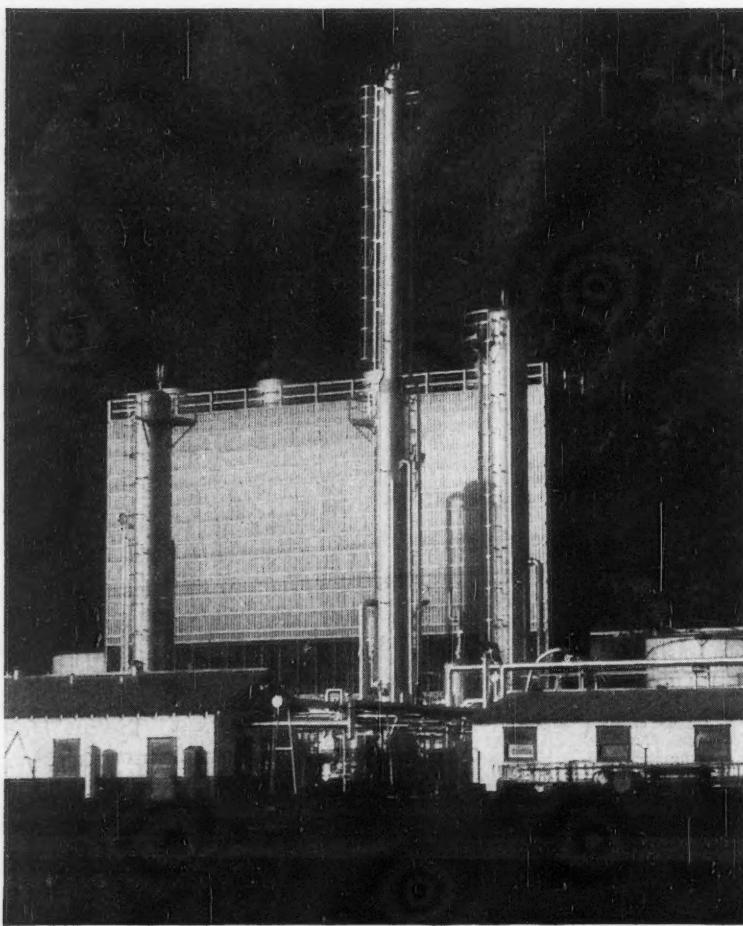


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## Cooling Towers —

(Continued From Page 10)

The time required for the entire operation is less than one man-hour per day.

### Laboratory Testing

The laboratory testing schedule consists of checking pH four times weekly and weekly tests for chromate and chlorides; pH, chloride and chromate tests are made on Fridays. No tests are made on Saturdays, Sundays or Mondays. This schedule has been ample.

The chloride test is used to determine concentration cycles. Weekly tests of make-up water have been satisfactory for adjusting to seasonal variations in water quality. Dissolved solids in the recirculating water vary from 5000 to 8500 ppm during the year, but because of the high cost of water (three times that paid in Los Angeles) the prime consideration is water savings rather than dissolved solids content of the water.

Maintaining concentration cycles with the old atmospheric towers was difficult because of heavy and highly variable windage losses. These towers and the present induced draft units were subject to abnormally strong winds. Acceptable control of the concentration cycles was obtained except when the wind came from the East, off the Mojave Desert. These winds generally blew during the dry season when water is scarce. This was one reason that Tidewater changed to induced draft towers which resulted in maintaining five to six concentration cycles, and significant water savings in this water-shy area.

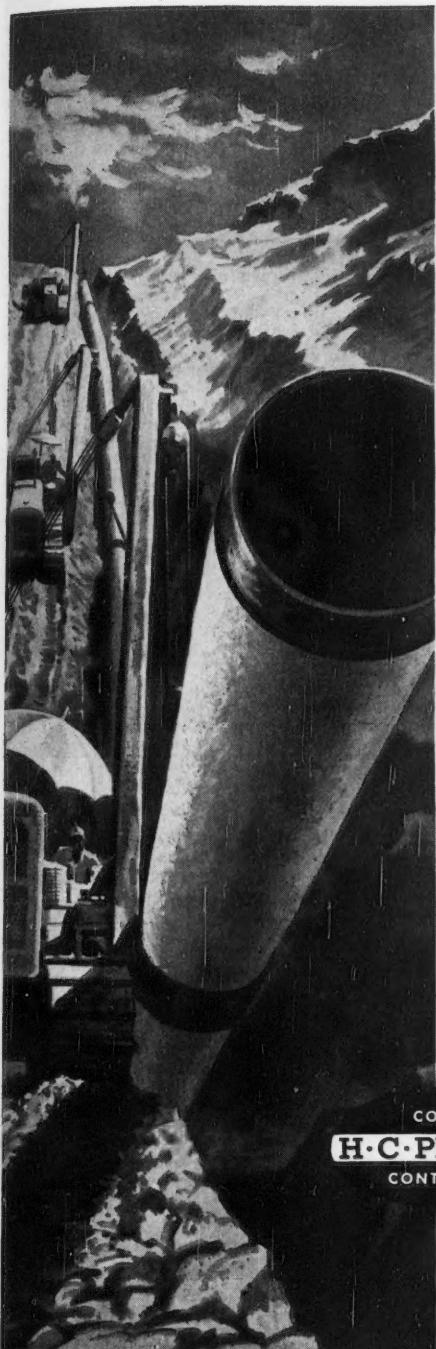
Retention of pH and inhibitor concentration within prescribed limits depends upon maintaining the same number of concentration cycles in each cooling system. The pH of the recirculating water is the result of the concentration cycles and the residual alkalinity in the make-up. When cooling water alkalinity is increased, the pH rises; when alkalinity is decreased, the pH is lower.

### Blowdown Control

Because made-up water is concentrated five to six times in the tower, the alkalinity and inhibitor entering with it also are concentrated. Therefore, make-up carrying a given quantity of alkalinity and inhibitor will have a higher pH and inhibitor concentration at six cycles than it would at five. It is impossible to maintain precise control; however, it is relatively easy to keep all tower waters at five to six cycles with some minor variations. For this reason, blowdown must be controlled as accurately as possible by inserting orifices in the blowdown line. The blowdown is taken from the return lines to the top of the tower (Figure 1). This assures constant pressure at the blowdown line which causes the blowdown orifice to discharge a constant water flow to the disposal system. A break in each blowdown line permits visual inspection of flow.

An orifice is inserted in a high-speed handlebar pipe union. The orifice is drilled through a stainless steel disk which fits into a socket machined into the union. A stock of interchangeable disks with different size orifices is maintained for use whenever a change in blowdown rate is required. A slightly

(Continued on Page 20)



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**Abstract**

Discusses unique water conditions found in tidal estuary at Port Alfred, Quebec, Canada, and their effect on cathodic protection of steel piling on wharves. Factors discussed include salinity gradient at various water depths, oxygen saturation and seasonal water freshening. Also considers effect of protection current in the tidal zone and depolarizing effect of tidal cycles. Discusses cathodic protection design considerations made on the Port Alfred pilings. 5.2.1

two other locations to confirm the reasoning that conditions found at Port Alfred are typical.

**Tidal Estuaries**

For the purpose of this article, a tidal estuary is defined as a relatively long and narrow channel where a fresh water river flows into a body of salt water. This will be a point where tidal action is still apparent, and, as at Port Alfred,

may have a considerable rise and fall. Because of the different densities, fresh water from the river will flow out over sea water for a considerable distance. Port Alfred is located about 400 miles from the Gulf of St. Lawrence and 70 miles from the St. Lawrence River which, at its junction with the Saguenay, shows a slight salinity gradation.

In spite of its distance inland from the Atlantic Ocean, Port Alfred has an extreme tidal variation of 24 feet, the average variation being about 12 to 13 feet.

As shown in Fig. 1, there is a variation in salt concentration with depth from a value of about 5 grams per liter chloride in the surface water to 24 grams per liter in the water below about 40 feet. Furthermore, there is a seasonal variation in the water's salinity down to 30 feet. During the months of May or June there is the annual spring run-off, caused by melting snow in the Saguenay water shed. This spring flood reduces salinity of the superficial water to about 1.2 grams per liter of chloride. This freshening effect has been found at depths of about 30 feet, almost to the

(Continued on Page 16)

## Current Densities to Protect Steel Piling in Fresh Water Tidal Estuaries\*

**Introduction**

BASED ON experience with cathodic protection systems on steel pile wharves at Port Alfred, Quebec, this article proposes that due consideration must be given to unique water conditions found in tidal estuaries. Data are given for two locations where fresh water rivers flow into sea water in estuaries which show a marked salinity gradation with depth of water. A seasonal freshening of the surface water is also noted. This freshening dissolves calcareous coatings, and the depolarizing effect of the oxygen saturated overlying water seems to demand cathodic protection current densities of about 10 milliamperes or more per square foot of steel surface instead of the accepted criterion of 5 milliamperes per square foot.

At Port Alfred, Quebec, on the Saguenay River, two large wharves are used for shipment of aluminum metal and paper, bauxite and other raw materials. One of these wharves, Powell Wharf, was built with vertical H-beam steel piles; the other, Duncan Wharf, was built with wooden piles which are being replaced with steel.

When driven, steel piles of Powell Wharf were given a coat of bituminous paint which failed in about a year, leading to corrosion of the steel. When the corrosion rate became apparent, a cathodic protection system was installed. Protection is included in the rebuilding of Duncan Wharf, based on the experience gained from the other wharf. In operation for about four years, the Powell Wharf protection system has given adequate protection although there have been some maintenance problems.

Several factors have lead to the belief that tidal estuaries are different from open sea water in respect to corrosion of long steel piles and that consideration must be given to these differences when designing cathodic protection systems for estuary installations. A certain amount of data has been obtained from

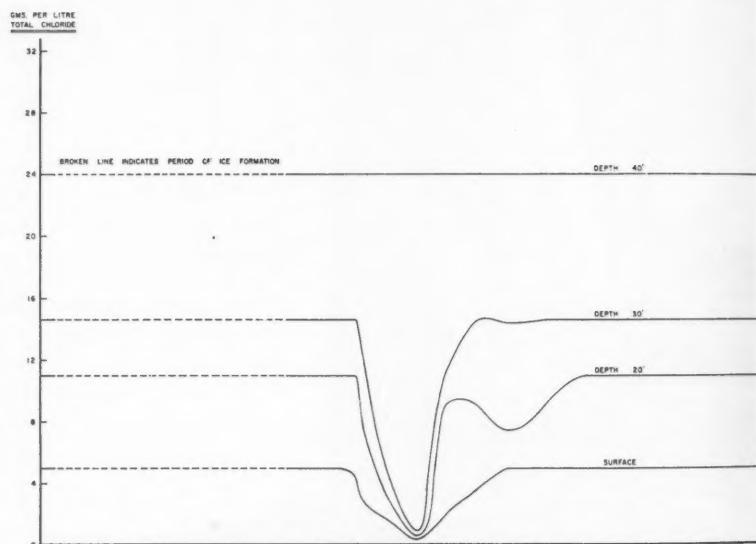


Figure 1—Graph showing seasonal variation of salinity with depth at Port Alfred, Quebec, Canada.

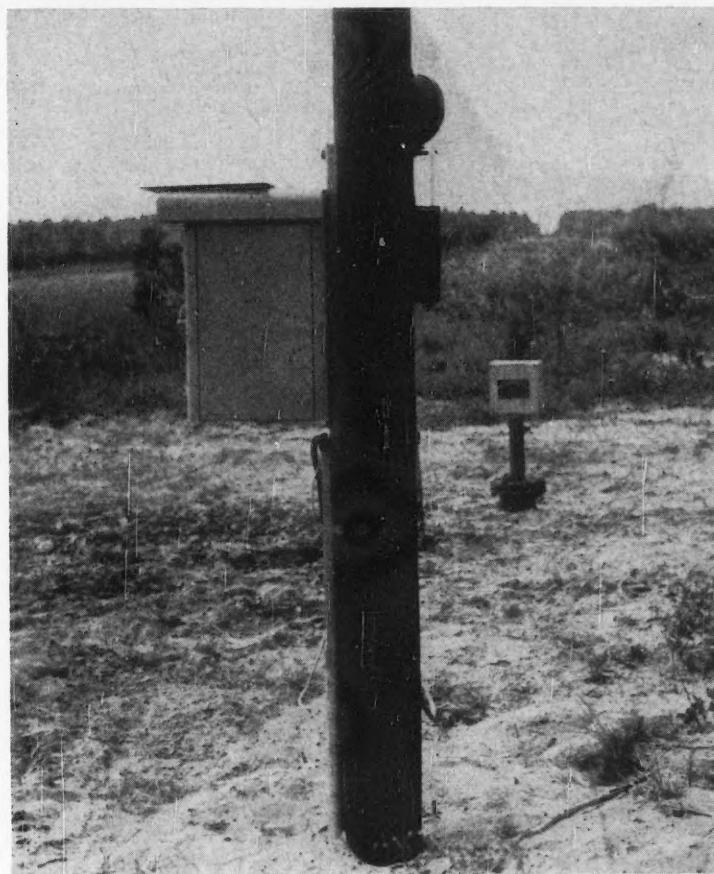
TABLE 1—Salinity at Various Depths in Rio Nunez, French West Africa

DATE (1958)	Tide Level	SALINITY (Grams Per Liter Total Solids)		
		Bottom	Half Depth	Surface
Aug. 18.....	Low	3.9	2.9	2.6
	Half Flood	6.0	5.7	4.2
	High	11.5	10.9	4.6
	Half Ebb	4.3	4.4	5.4
Aug. 19.....	Low	2.8	0.9	0.7
	Half Flood	6.6	6.2	4.2
	High	12.3	8.9	5.1
	Half Ebb	5.6	5.4	5.3
Aug. 20.....	Low	1.9	2.5	2.6
	Half Flood	2.0	1.9	1.9
	High	11.3	10.5	3.9
	Half Ebb	3.8	4.2	3.7
Aug. 22.....	Low	1.1	1.0	1.2
	Half Flood	6.4	2.4	2.0
	High	8.1	5.9	3.6
	Half Ebb	3.0	3.2	3.4
Aug. 23.....	Low	10.2*	1.3	0.7
	Half Flood	10.2	3.9	3.9
	High	8.7	6.5	3.9
	Half Ebb	3.6	3.9	3.9

\* Suspected analytical error.

\* Revision of a paper titled "Some Notes on Cathodic Protection in Tidal Estuaries" by D. B. Bird, Tidewater Oil Co., Inc., Delaware City, Del., and H. G. Burbidge, Aluminum Company of Canada, Limited, Montreal, Canada, presented at the Eastern Division, Canadian Region, National Association of Corrosion Engineers, January 12-14, 1959, Montreal, Canada.

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## Current Densities—

(Continued From Page 14)

same degree as found at the surface. As Figure 1 shows, there is little or no freshening of water below 30 feet.

There is a corresponding change with depth and with season in the water's conductivity. Superficial conductivities range from 65 ohm cm to 400 ohm cm over the annual cycle, whereas at depth the conductivity is 25 ohm cm. Being fresh river water, the superficial water is saturated with oxygen and acts as an excellent depolarizer. There is a lack of oxygen in the deeper water, as evidenced by the discovery of highly anaerobic conditions in the loose, largely organic muck on the bottom and in the dense clay that constitutes the bottom.

Salinity measurements also have been

made at the mouth of the Rio Nunez at and near Port Kakande, in French West Africa. Here a fresh water river flows into the Atlantic Ocean through a narrow channel but on a coastline which is relatively little above sea level and which is characterized by extensive tidal flats. During the 10-month dry season, the salinities at the river mouth and part of the way up the river are almost constant at 3.4 to 3.7 grams per liter at any depth. During the rainy season, there is such an excessive flow of fresh water in the river and across the tidal flats that the salinity is reduced to approximately 0.4 to 1.5 grams per liter.

Table 1 shows some typical results of analyses. The samples were all taken at one station where the bay bottom is at a depth of minus 15 feet relative to mean low tide at zero. At high tide the

bottom salinity is generally much higher than at any other level, or even at the same level at mid and low tides. This conforms with the hypotheses developed from Port Alfred data that stratification of the fresh and saline waters is substantially the same at high and low tides. When a proposed wharf is built at the mouth of the Rio Nunez and dredging is carried out to permit ships drawing 28 to 30 feet to berth at the wharf, there will be a gradation similar to that at Port Alfred, but only during the rainy season.

An extensive survey of the River Tees in England also showed gradation in salinity with depth and seasonal freshening.<sup>3</sup>

From this information on estuaries, the salinity gradient and freshening of superficial water can be assumed characteristic of any estuary where a fresh water river of any considerable size flows into sea water. This may not be true if the fresh water in flow is too small relative to the estuary's size for the stratification conditions to develop. Saguenay River's fresh water flow is large and averages 54,000 cfs. Spring flood flow is about 250,000 cfs.

### Significance of Estuary Conditions

In a tidal estuary, five factors affect the nature of the corrosion of vertical steel piles and cathodic protection used on the piles. The factors are salinity gradient, oxygen saturation of the surface water, seasonal freshening of the water and anaerobic condition found in the deep water. A fifth factor (found when a harbor of any considerable activity exists on an estuary) is the film of oil and contamination on the surface of the water resulting from pollution. Only three of these factors will be discussed here.

### Salinity Gradient

Salinity gradient produces a concentration cell along the length of vertical piles. The corrosion effects induced by this gradient alone are not too significant when compared with the differential aeration cell between the exposed steel in the saturated surface water and the permanently immersed surfaces in the more oxygen free deep water.

### Oxygen Saturation

Adding to the differential aeration cell that would normally exist in a body of water is the oxygen saturation of the fresh water flowing into an estuary. This augments the depolarizing effect in the tidal zone, keeping the steel in this area cathodic to steel that is immersed continuously. Also, the anodic effect falls off with depth as the salt concentration increases, thereby making the steel at depth in effect cathodic to the steel in the mean low tide area. This produces rapid corrosion in the ± 5-foot zone at extreme low tide.

In estuaries, the pattern of corrosion on steel piles differs somewhat from corrosion in natural sea water. Figure 2 shows a comparison of the corrosion patterns at Port Alfred and in natural sea water. The familiar curve is given for relative loss in metal thickness at varying depth at Kure Beach, N.C.<sup>2</sup> For comparison, typical potential gradient curves are given on the piles at Port Alfred. These are called typical because there is considerable variation from pile to pile.

### Seasonal Water Freshening

The third factor affecting steel piling corrosion is seasonal freshening of the upper water. Not only does this increase

(Continued on Page 18)

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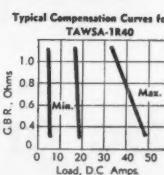
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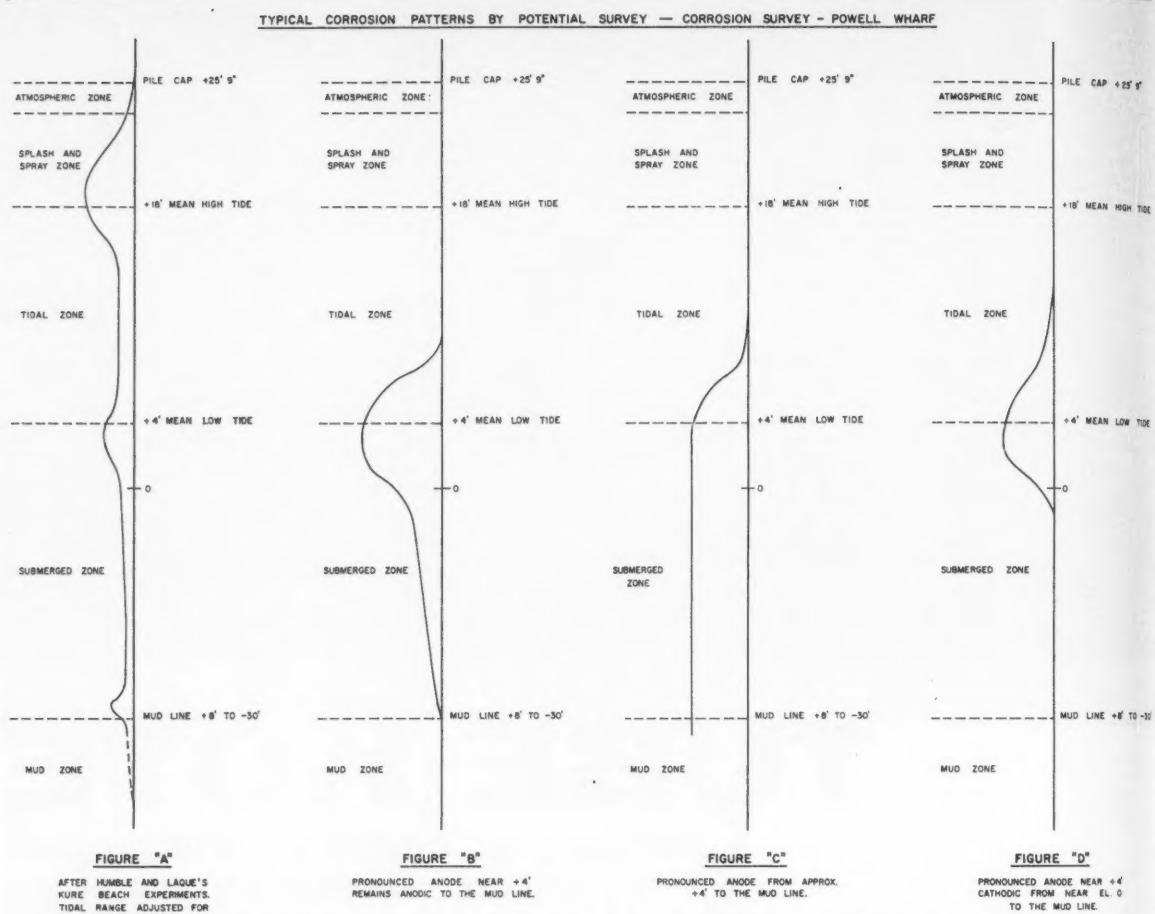


Figure 2—Comparison by potential survey of corrosion patterns at Port Alfred and in natural sea water.

## Current Densities —

(Continued From Page 16)

the depolarizing effects of the superficial water, but it dissolves calcareous coatings deposited by the effect of cathodic protection currents during the remainder of the year. This happened with design protection currents of about 5 milliamps per square foot of steel surface. Evidence indicates that it would happen even with current densities of 8 to 10 milliamps per square foot. Evidence also indicates that cathodic protection of clean, unpainted steel piles can be maintained only in the fresher water zones with currents of about 10 milliamps per square foot or higher. Success obtained so far at Port Alfred with design currents of 5 milliamps per square foot for the water zone and 1 milliamp per square foot for the mud zone is attributable largely to remains of the original paint coating and the film of Bunker C oil that formed on the piles following the accidental discharge of a considerable oil from a tanker berthed at the wharf. After this oil film washed away and the paint coating deteriorated, incipient corrosion of the piles has occurred. Consideration is being given to the need for increased current.

### Tidal Zone Protection

In a study to determine the effect of protection current in the tidal zone and  
(Continued on Page 20)

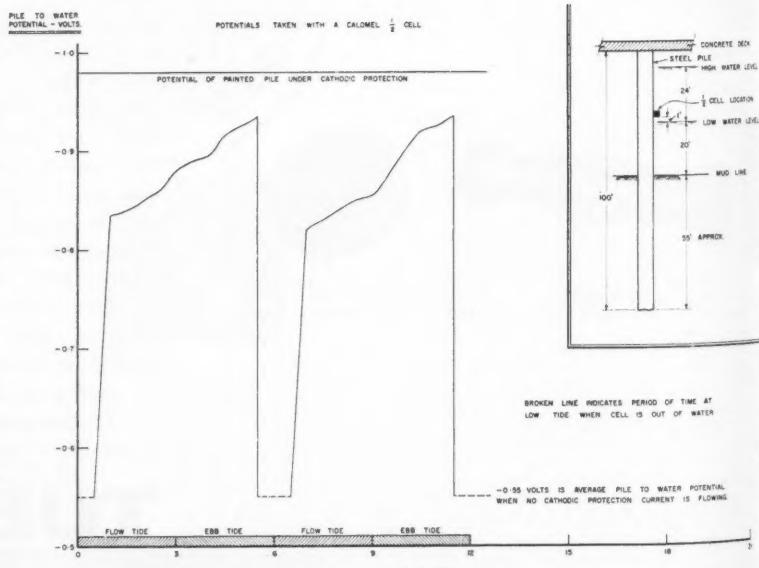


Figure 3—Plot of potentials of the pile relative to sea water, taken with a calomel half cell at Port Alfred, Quebec, Canada.

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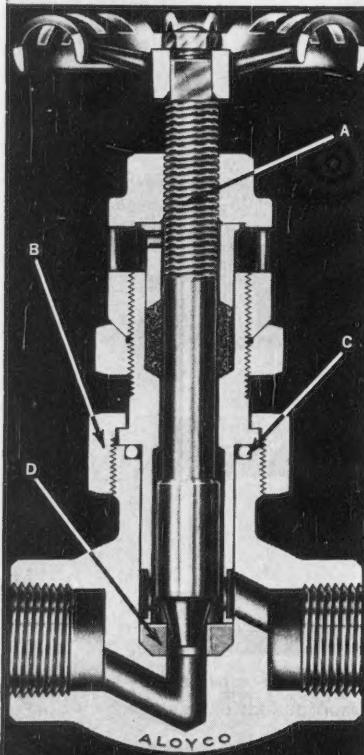
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0.4



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## **Current Densities—**

(Continued From Page 18)

the depolarizing effect of rising and falling tides, a calomel half cell was suspended near a pile at an elevation just above extreme low tide. The cell was immersed as the tide rose and remained so until the ebb-tide had fallen below the elevation of the cell.

Figure 3 is a plot of the potential of the pile relative to sea water. The potential decrease from the -0.55 volts that is normal for this wharf without the protection current to a value of about -0.82 volts in the first 30 minutes. As the pile remained submerged, the potential fell to -0.93 volts, and then, of course, increased sharply when the half cell was exposed. This plot shows that a relatively good protective environment can be maintained during the tidal cycle on submerged steel in the tidal zone. Though the time of submersion is much shorter, a protective potential is established over the time of submersion of the piles at highest tide levels. Also shown is the potential for a painted pile. In this latter case the depolarizing effect of the pile's exposure to the atmosphere is not apparent. The potential at the moment of submersion was -0.98 volts.

Results of this test show that protection can be maintained in the tidal zone, using only the applied protective currents.

### **Design Considerations**

Design of the cathodic protection on the two Port Alfred wharves was based on a current density requirement of 5 milliamperes per square foot of steel exposed to the water and 1 millampere per square foot of steel in the mud zone. The system has given satisfactory protection to Powell Wharf, but this is partially attributed to the oil film on the piles in the tidal zone. This oil reduces the steel area exposed in the tidal zone, therefore concentrating the current to small non-coated areas of the piles. Current density on the areas not covered by residual paint and oil films therefore must be higher than the 5 design current density.

Actual measurements of current density would be difficult and have not been attempted. The oil film is wearing off, and some active corrosion has been observed on a protected test pile that is periodically removed for examination. Cathodic protection on Duncan Wharf piles which have no paint or oil film also was designed on the basis of 5 milliamperes per square foot of exposed steel in the water zone. This was inadequate and did not stop the corrosion.

From experience with the two wharves, a minimum current density of 8 milliamperes per square foot of steel in the water area is required. A safe design basis would be 10 to 12 milliamperes per square foot of steel in the water zone and 1 to 3 milliamperes in the mud zone. Current density requirements in the mud zone vary with mud resistivity, presence of anaerobic bacteria, soil stratification, etc.

Current density of 10 to 12 milliamperes per square foot of exposed steel in the water zone is intended for unpainted steel. This value, of course, could be reduced if the steel were painted. Normally, the initial current density used for cathodic protection can be reduced as polarization and calcareous coatings form. Calcareous coatings are removed rapidly under spring flood conditions; therefore no cut-back in cathodic protection current can be made. A cut-back

in the current might be made as the surface water salinity content increases during the summer months. However, this would not be advisable because, even during the maximum salinity months, surface water always is oxygen saturated and considerably less saline than the water at depth. No tests have been made in varying the current density with seasonal variations. Perhaps this would be a worthwhile investigation.

In a tidal estuary where corrosion is concentrated in one zone, it would be ideal to locate the anodes close to the corroding zone. This might permit use of less total current by virtue of the current density's tendency to be higher on the steel nearest the anodes. Unfortunately, Port Alfred's severe winter icing conditions necessitate anode placement in as deep water as possible, and where the bottom is exposed at low tide they are buried in the mud. This was necessary to avoid serious icing damage to anodes and wiring.

### **References**

- H. W. Harvey, Chemistry and Fertility of Sea Water, Cambridge University Press, 1957.
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### **Cooling Towers—**

(Continued From Page 12)

larger or smaller orifice can be installed in the union in a few minutes. Orifice changes are made about 10 or 12 times per year in the entire system due to weather conditions and changes in heat load. The object is to maintain the same number of concentration cycles rather than regulating the dissolved solids in the recirculating water.

Tests for pH serve more than one purpose. If there is much pH variation in one tower, the blowdown should be adjusted. The test's primary purpose, however, is to check on the acid feed pump.

Biofouling has been controlled by application of a commercial biocide every other week. This is added to each tower basin rather than through the make-up line. The reason for this is that the biocide requirements are not the same for each tower because of differences in heat load. The commercial biocide is supplemented by chlorine which is added to the tower over-shot two or three times a year, as required.

### **Conclusions**

The central feed system for acid and inhibitor has been successful. It is simple and requires a minimum of labor. Like any chemical process, it requires a "break-in" period for the operators, after which it becomes routine. No serious difficulties have appeared after eight years of operation. Replacement of parts subject to acid corrosion have been less than anticipated. Success has been due, in a large part, to cooperation of plant personnel.

Central feeding has eliminated the problems of batch feeding with lax operations. Scale under former feeding often built up so manual chipping had to be done at least once a year. Still overhead heat exchangers had to be opened and cleaned every six weeks. Today there is no scale. Still overhead heat exchangers run two years before inspection is necessary. Flushing with water only removes silt accumulations. Corrosion has been controlled satisfactorily as shown by test coupons, inspection of equipment and replacement record. There has not been a failure of equipment due to water side corrosion.

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Colorado River water that is used in Southern California is hard on metals — particularly bronzes with high zinc or aluminum contents.

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Type A Ni-Vee Bronze, with a zinc content of 2%, is well under the threshold at which dezincification

will occur in the Colorado River water. What's more, the composition of Nickel, tin and copper in this alloy makes it highly resistant to corrosion and the effects of velocity. The metal is also responsive to heat treatment to obtain higher than ordinary mechanical properties. In this instance, 85,000 psi tensile strength and 180 Brinell hardness were easily obtained.

### 5 types of Ni-Vee bronze provide a range of desired properties

Altogether there are 5 basic types of Ni-Vee bronzes, all heat treatable, all with excellent corrosion resis-

tance. Ni-Vee A provides extreme strength; Ni-Vee E provides extreme resistance to galling. The other types have desirable pressure and bearing properties.

For information that will help you specify the best type of Ni-Vee bronze for a particular part, simply write for "Engineering Properties and Applications of Ni-Vee Bronzes," and the Ni-Vee Bronze "Buyer's Guide," listing sources of supply.

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**Abstract**

Describes an electroless process for chemical nickel plating. Gives properties of the coating and discusses its applications for corrosion or product contamination prevention, hard coating, soldering or brazing aluminum or stainless steel, prevention of galling tendencies in metals and prevention of stress corrosion in stainless steel.

5.3.4

**Introduction**

**A**N ELECTROLESS process for chemical nickel plating is the culmination of research beyond the U. S. Bureau of Standards' work in 1947,<sup>1</sup> which discussed the large amount of seemingly unrelated information surrounding the reaction between hypophosphite and a nickel salt.<sup>2</sup> Additional research has resulted in a more stable reaction with relatively rapid rate. Thus techniques have been developed by which nickel plating of large objects such as interior coatings for tank cars can be achieved economically.

**Process Description**

Chemical nickel plating, registered by the General American Transportation Corporation as the Kanigen process, is a means by which a nickel phosphorous alloy can be applied as a coating to a variety of metals and in some cases to non-metals.

When an iron object is introduced to a chemical nickel-plating bath, the first instantaneous reaction is a displacement resulting in nickel deposition on the iron surface due to the relative electro-chemical potentials. The nickel so deposited then acts as the catalyst for the catalytic dehydrogenation of the hypophosphite ion to meta-phosphite ions with the liberation of atomic hydrogen.<sup>3,4</sup> This atomic or nascent hydrogen is the direct means of reducing nickel from its salt as a metal coating. This reaction results in the final liberation of molecular hydrogen as gas bubbles. At the same time that this is going on, the meta-phosphite ion reacts with water to form orthophosphate.

With this nickel reduction, a side reaction occurs between hypophosphite and probably phosphite ions, the atomic hydrogen and the nickel cations. This all results in formation of nickel phosphide and accounts for the presence of the phosphorus in the final coating. These various reactions are pH dependent, and an increase in pH causes a decrease in the rate of this last reaction. This results in a lower phosphorus content for platings obtained from alkaline baths.

Because the over-all deposition reactions are pH dependent, the pH must be controlled in order to achieve a constant and continuous rate of deposition. The logical control in a batch process is the use of buffering salts. Also in a batch operation, hydroxyl ions can be added and, at suitable intervals, nickel and hypophosphite can be added, thus making a continuous operation.

This procedure seemingly solves the problem of continuous plating by chemical reduction means. However, during plating, reaction products are being built up in the form of phosphite. Nickel phosphite is relatively insoluble and, to

make matters worse, has an inverse solubility curve. In the pH range where catalytic reduction is an optimum for acid solution, nickel phosphite has a solubility of about 0.07 mols per liter. When the phosphite concentration goes beyond this, precipitation of nickel phosphite takes place. This material comes down as a finely dispersed semi-colloidal solid which can absorb active hydrogen. After some time, the suspensoids act as catalytic nuclei causing coating roughness and finally bath decomposition.

Recent research has succeeded in raising this phosphite-concentration limit above one mol per liter by use of certain selected nickel-chelating compounds. These compounds must be selected carefully for their nickel complex stability constant.

This line of attack, however, slows up the over-all rate of nickel deposition even though stable baths are obtained. Further work resulted in the discovery of organic compounds which would act

steel. When steels, heat-treated to strength levels above 170,000 psi and containing two parts per million or more of hydrogen, are loaded at about 150,000 psi in the presence of a notch, failure occurs in a measurable time. If the hydrogen content is below critical, the steel will sustain the load indefinitely.

A statistical study was conducted on chemical nickel plated SAE 4340 steel heat treated to 200,000 psi and 260,000 psi. No significant reduction in tensile properties was noted due to the plating process. Also the plating did not significantly alter capacity for impact energy absorption. The electroless plating plus hydrogen relief, however, reduced the fatigue-endurance limit of reverse-bend specimens by 25 percent.

Each case involving fatigue should be evaluated independently as to whether or not plating can be tolerated. Many factors are involved in a given part's fatigue properties, such as geometry of the part, type of loading and heat treat-

## Corrosion Control Applications of **Electroless Process for Chemical Nickel Plating\***

as rate increasers or exaltants. These exalting compounds are generally short-chain aliphatic dicarboxylic acids.<sup>5</sup> Action of exaltants is often dramatic, such that over-all rate increases of ten times have been experienced. Some ordinary baths with low rates of about 0.1 mil per hour will exhibit a rate close to 1.0 mil per hour on the addition of sodium succinate.

**Properties of the Coating**

The resulting chemical nickel alloy is unique in a metallurgical sense—a metal in the amorphous state. X-ray diffraction patterns of as-plated electroless nickel alloy are similar to those obtained for glass and plastics.<sup>6</sup>

The nickel phosphorus alloy can be made to crystallize rapidly on heating to temperatures of 400°C and above. As plated from an acid bath, the material has a phosphorus content from 8 to 10 percent and a hardness of about 50 Rockwell C. This hardness can be raised by controlled heat treating to about 64 Rockwell C. Also, close control of the desired region of the heat-treating curve gives added toughness with the increased hardness. These properties are useful in many machine applications where hard coated surfaces are needed.

Studies have been made on plating's effects on fatigue life of high tensile steels.<sup>7</sup> Dissolved hydrogen causes considerable deterioration in certain mechanical properties of high strength steels. This deterioration is generally manifested in a serious reduction in the metal's fatigue life. Hydrogen in excess of two parts per million, especially when it is in the range of about ten parts per million, seriously reduces the tensile strength and ductility of high strength

ment given the base alloy. A two percent total strain under load can be supported by a one-mil thick coating of this nickel plating without danger of cracking. When the plating is structurally transformed by heating at about 700°F for one-quarter of an hour, a strain of 6 percent can be supported.

Resistance of electroless nickel to corrosive environment parallels that of ordinary nickel. In general, wherever nickel is a desirable metal for use, electroless nickel provides comparable chemical corrosion resistance.

Nickel applications can be divided into two categories: (1) Where nickel is the metal dictated by the corrosive environment and no chances can be taken with the possibility of product contact with the base metal. (2) Where nickel is the metal dictated by corrosive environment but minor contact with the base metal is not a serious consideration.

In the first category, only solid alloys are to be considered. Clad steel and plated steel are ruled out because probabilities are high that some defect can be found in any coating. These probabilities increase with the area coated.

The second category includes the numerous applications for chemical nickel coating. Of major importance are applications involving no actual corrosive attack on nickel but dictating the use of nickel merely to prevent iron contamination. These are to be found in many industries such as edible oil processing, detergents, caustic soda and colorless ingredients for use in manufacture of various synthetic resins.

Outdoor exposure tests on electroless nickel have been conducted for about

(Continued on Page 24)

\* Revision of a paper titled "The Kanigen Process for Chemical Nickel Plating With Applications to Petrochemical and Allied Equipment" by W. J. Crehan, General American Transportation Corp., Chicago, Ill., presented at the Western Division Conference, Canadian Region, National Association of Corrosion Engineers, Feb. 11-13, 1959, Calgary, Canada.

## Electroless Process—

(Continued From Page 23)

two years. In general, this testing is for informative purposes only because industrial platings are rarely used for protection against outside exposure. Paints are more economical to use than metallic coatings.

These outdoor aging tests have indicated some peculiarities in this type of plating that might otherwise have been unnoticed. One peculiarity was that panels not heat treated failed more rapidly than heat-treated panels. The panels were identical except for the heat treatment.

Investigation showed that base metal discrepancies cause growth faults in the coating which in some cases can become focal points for corrosive attack. Where corrosive attack occurred, it occurred at a growth fault. However, all growth faults were not sources of corrosive attack. In the heat-treated panels, these growth faults were no longer visible in micro section and apparently were healed by fusion or stress relieving in the heat-treating process.

Another peculiarity noticed was that of the rapid failure of panels that had been electroless plated with chromium plating on top. Electroless plating of itself showed much better resistance to outdoor exposure than electroplated panels. When chromium was added, the picture was completely reversed in that the chromium-plated electro nickel was much better than the chromium-plated electroless. This can be understood when one knows that the as-deposited chemical nickel plating is highly-stressed. Also, chromium plating is a highly-stressed coating. When the two are superimposed, the result is cracking of the total plating, exposing base metal.

### Applications

The many interesting applications for chemical nickel plating can be classified for discussion according to the primary reason or purpose of the plating, as follows: (A) corrosion protection or product contamination protection, (B) hard coating, (C) soldering or brazing of aluminum or stainless steel, (D) prevention of galling tendencies in metals and (E) prevention of stress corrosion in stainless steel.

### Corrosion Protection

Electroless nickel is being used as a tank car lining to provide product protection for caustic soda, ethylene oxide, tetraethyl lead, tall oil and glucose. About 75 plated cars have been in satisfactory operation for a year or more.

Recently, a Louisville rotary dryer (8 ft by 30 ft) and a rotary reactor (8 ft by 45 ft) were fabricated from carbon steel and coated with three mils of chemical nickel plate. This equipment was built for the processing of a non-corrosive foodstuff additive. The nickel lining was applied solely to prevent iron contamination which would result in product discoloration.

In missiles, many plating applications are made for prevention of corrosion while parts are in storage. In most of these cases, the plating itself is of no value to the proper functioning of the part in flight. Example of this was the chemical nickel plating of the fuel injector plates which were used in the Explorer satellite. These fuel injector plates had been in storage for two years before the launching. The Atlas satellite had chemical nickel plated fuel injector nozzles in each stage.

Chemical nickel plating has been used on the internal surfaces of many high pressure gas storage bottles used in ground installations for launching liquid fuel rockets. This plating again is applied to prevent corrosion that might plug small orifices. This same type protection is used in the handling of jet aircraft fuel. Chemical nickel plating has been applied to the fuel handling pumps on naval aircraft carriers and fuel metering devices on jet aircraft.

Aboard aircraft carriers, an aqua system of fuel handling is used. While refueling operations are being carried out, sea water is added to the storage tanks to eliminate the possibility of developing an explosive vapor mixture.

The Petroleum Branch of the U. S. Army Engineer Research and Development Laboratories Corps of Engineers report that 0.001-inch electroless nickel on steel pump shafts will prevent damage normally caused by products being pumped plus protection from moisture during idle periods. It is concluded that chemical nickel plating saves on critical materials without sacrificing function. This test work led to the inclusion of electroless nickel plating in a military specification.<sup>8</sup>

Chemical nickel plating has been used successfully in the petroleum industry by a large West Coast pump manufacturer. Plated carbon steel castings were used to replace alloy steels for considerable cost savings and good service life. On heavy casings these savings can be about 75 percent.

These pumps are handling many petroleum products, some of which contain abrasive particles found in sludges. These products quickly erode the inner surfaces of unprotected pumps, seriously impairing their function.

A typical case is the bottom pump of a fractionating tower. This pump transmits a very heavy, hot oil which frequently contains highly abrasive substances in suspension. Because of intricate shapes in the internal pump parts, electrolytic plating with its resultant unevenness would not offer enough abrasion resistance to give a pump any practical service life, so chemical nickel plating is used. The alternative is to build the entire pump of costly high alloy steel.

Three to five-mil coatings of electroless nickel have been used on special purpose valves by a company specializing in petroleum service valves for handling material with hydrogen sulphide content. Field reports have been good; several valves have been placed in service.

Electroless nickel plating is of value in equipment for manufacturing polyethylene or poly propylene—particularly valves, tanks, etc. The plating of internals of a butyl rubber dissolver has successfully protected the product and has shown a considerable saving in original equipment.

### Hard Coating

Electroless nickel plating has been used to provide a hard surface coating or a supplemental coating applied to alloy steels which enables the surface skin to be hardened to a degree higher than is possible to attain in the base metal. Such applications occur in rotatting and reciprocating machinery such as gas compressors, pumps of various types and hydraulic cylinders such as are used in aircraft actuating mechanisms.

A hardened surface of chemical nickel plating has a hardness range close to that of chromium and also wear characteristics comparable to chromium.<sup>9</sup>

Chemical nickel plating has been applied to aluminum sheaves to provide a hard wear resisting coating, where aluminum must be used because of its light weight, and yet must have a hard wearing surface.

In the manufacture of electrical components, chemical nickel plating in the coating of certain relay armatures has given increased service life. Also, the coating is non-magnetic, thus making it possible to reduce the air gap and consequently cut oxidation due to arcing. Some minor corrosion protection is obtained because the coating is applied usually over iron.

### Soldering or Brazing of Aluminum or Stainless Steel

In the electronics industry, aluminum often must be soldered. Because it is almost impossible to solder directly to aluminum, this operation is facilitated by use of a thin nickel flash. Because electroless nickel can be plated directly on aluminum, this new plating process has solved difficult production problems of plating over intricate shapes used in electronic devices. A thin coating of chemical nickel plating over stainless steel will facilitate brazing operations used in fabricating items such as gaseous heat exchangers.

### Prevention of Galling Tendencies in Metals

Many materials, for example, stainless steel, aluminum, titanium, etc., show a tendency to gall when used with mating parts made from the same material. This tendency can be eliminated by application of a relatively thin coating of electroless nickel. Threaded stainless steel pipe fittings are improved in their operation by plating one of the members. In the manufacture of titanium parts that must be threaded or for parts that move against each other, nickel plating is a necessity in preventing titanium's galling tendency.

Many hydraulic actuating cylinders fabricated of titanium for missile applications have been chemical nickel plated.

### Prevention of Stress Corrosion in Stainless Steel

Application of a nickel plating to an object made of stainless steel may seem peculiar, at first. In actual use, however, there are many cases where stainless is a necessity for handling corrosive commodities. In coolers where the water on the cooling side has a relatively high chloride content, stress corrosion cracking in the stainless steel becomes a serious maintenance problem.

This type corrosion can be prevented by presence of a good favorable electrolytic couple, such that the stainless is protected by another metal, e.g., nickel. Due to the involved geometry of such pieces of equipment, chemical nickel plating usually is the only type of plating which will successfully provide the intimate coverage required for protection.

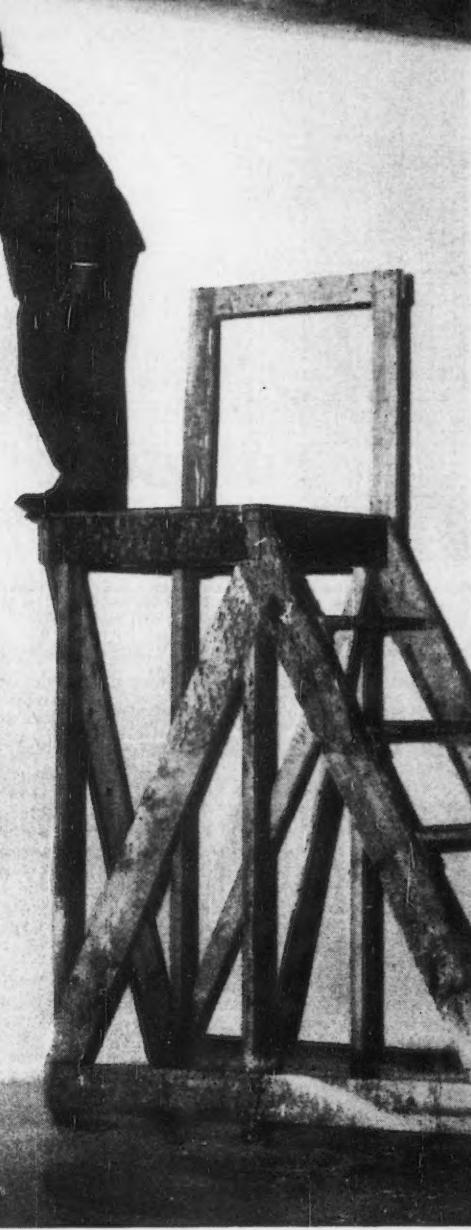
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# CORROSIONEERING WITH LAMINAC® POLYESTER RESINS

**Two years  
in formaldehyde  
— reinforced  
Laminac lining  
surpasses its  
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This test fully confirms the faith LAMINAC inspired 31 months ago when this 200,000-gallon tank was coated with Cyanamid's polyester resin. Since then the tank has held a 37% solution of formaldehyde at 90°F. without corrosion even at the unprotected ceiling line.

The lining is fabricated of glass cloth and LAMINAC Polyester Resin 4109, with a clear finish coat of LAMINAC Resin 4111. It was installed on May 20, 1957, replacing a vinyl-based lining, the service life of which had been shortened to a point where the underlying concrete was deteriorating, and contaminating and discoloring the formaldehyde.

The results are also in on another stiff test given to LAMINAC! Starting Dec. 20, 1958, 200,000 gallons of a 44% solution of formaldehyde were placed in a tank at 120°F. In addition, the ceiling was coated to prevent corrosive effects. The solution remained in the tank for 1 year.

At the end of 1959, inspection revealed no breakdown or blistering, pitting, bleaching, cracking or pulling away from the LAMINAC-lined concrete wall!

The reinforced LAMINAC linings for these huge formaldehyde tanks are expected to last indefinitely.

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## Development and Characteristics of

# Reinforced Furane Resin Systems Used in the Chemical Industry\*

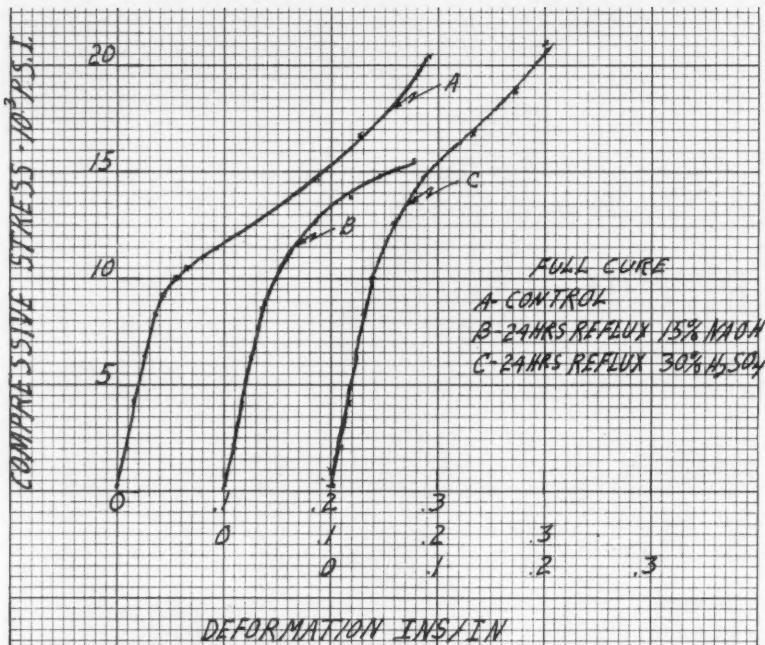


Figure 1—Chart showing stress-strain characteristics of a furane resin. Curve A is the control, Curve B for 24 hours reflux in 15 percent NaOH, and Curve C for 24 hours reflux in 30 percent H<sub>2</sub>SO<sub>4</sub>.

### Abstract

Discusses recent developments in curing of furane resin systems for use in chemical industry. Briefly describes chemical derivation of furane resins from furfuraldehyde. Gives physical characteristics of these resins and their applications.

**RECENT DEVELOPMENTS** in furane resins have made possible tighter, more complete cures, resulting in higher strengths than hitherto achieved by furane resins in reinforced plastic structures.

### Derivation of Furane Resins

The furane resins are derived from furfuraldehyde which is extracted from corn cobs, rice hulls, bagasse and other vegetable products rich in pentosans. Through hydrogenation, furfuraldehyde is converted to furfuryl alcohol and tetrahydrofurfuryl alcohol.

Furfuryl alcohol is the most important ingredient in the formation of resins, reacting vigorously in the presence of acids to form infusible thermosetting polymers. Sometimes co-reaction is carried on in the presence of formalde-

hyde or furfuraldehyde to form commercial synthetic resins. Furfuraldehyde likewise can be reacted with ketones or with phenols to form resins. However, furanes resins are generally considered synonymous with the polymers of furfuryl alcohol.

For a more complete understanding of furane resin behavior in the manufacture of chemical industry equipment the character of the polymer formation should be reviewed. As furfuryl alcohol polymerizes in the presence of strong acid catalysts, a condensation polymerization reaction ensues with the formation of water. This reaction is highly exothermic and will go out of control unless adequate safety measures are introduced. The resin increases rapidly in viscosity, followed by the formation of a weak gel. There is no so-called stage "B" involving a fusible and soluble resin; there is only viscous liquids or irreversible gels.

With continued curing, the gel forms a hardened solid, as a casting or as a laminate. If the curing proceeds too rapidly, strength is lowered and the friable character of the resin is more apparent. Another distinguishing feature is the apparent hardening from the outside in, as if air has a salutary effect. Until recent developments in furane resins,\* the completion of cure was often

time consuming or questionable. Inclusion of large amounts of filler, on the other hand, have helped stabilize furane systems by reducing shrinkage and offering practical measures for curing the material in situ.

Completeness of cure is important if furane resins are to be exposed successfully to corrosive chemicals. In most instances, room-temperature cure is not sufficient; high temperature post-cure is a must, and frequently long exposure to high temperature is necessary. Otherwise, chemical resistance of reinforced furane resin structures is decreased. A detailed review of furane resin chemistry is given in a book by Dunlop and Peters.<sup>1</sup>

### Physical Characteristics

Physical properties of cured furane resins are influenced in part by the catalysts used, cure conditions and reinforcements used. The reinforcements are important to chemical equipment because of the added mechanical strength. Materials widely used include carbon, graphite, acid-washed asbestos, silica and glass fibers. When woven glass and asbestos fibers are used, resin and catalyst are blended and applied to the cloth or batting in siter. As cure takes place, preferably in the presence of air, the structure is built-up to the desired thickness for strength and chemical resistance.

Typical properties of acid-catalyzed, unfilled, furane resins are shown in Table 1. These characteristics were determined on a commercial furane resin system. The data are presented to show the innate characteristics of a well cured furane resin. When combined with adequate reinforcement, such mechanical characteristics as impact strength and stiffness necessary for chemical equipment are realized. Data presented are in accordance with the latest test methods of the American Society of Testing Materials.

A comparison chart (Figure 1) shows the stress-strain characteristics of a furane resin as cured, after refluxing for 24 hours in 30 percent sulphuric acid and after 24 hours in 15 percent sodium hydroxide solution.

Properties of the cured furane resin depends upon the completeness of cure. Indicative of this would be hardness and shrinkage values of a typical furane resin system given in Table 2.

The effect of fillers added to the resin system vary, depending on their physical nature. Large loadings of asbestos contribute much to the strength though the chemical resistive qualities are basically dependent upon the furane resin. Pourability of the casting resin is decreased markedly as the asbestos fibers are added to the point where the materials must be applied by trowelling techniques. Shrinkages during cure are decreased markedly, almost a function of the percentage asbestos added.

Because of the current interest in glass cloth reinforcements, glass cloth laminates were prepared with proprietary furane resins and tested according to ASTM standards. The glass fabrics described in Table 3 were the wide open mesh fabric used for construction purposes and lining tanks. Resin contents were about 45 to 50 percent by weight. Flexural strength values of 28,000 to 29,000 psi reported for the furanes compare favorably with the 30,000 to 32,000

(Continued on Page 28)

\*Revision of a paper titled "Reinforced Furane Resin Equipment for Chemical Industry" by John Delmonte, Furane Plastics, Inc., Los Angeles, Cal., presented at the 15th Annual Conference, National Association of Corrosion Engineers, Chicago, Ill., March 16-20, 1959.

<sup>1</sup>Jet-Kote X-8, manufactured by Furane Plastics, Inc.



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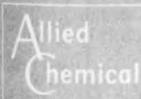
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TABLE 1—Physical Properties of Furane Resin\* (No Fillers)

Density g/cc.	1.29
Ultimate Compressive Strength.	19,000 psi
Flexural Strength.	5,000 psi
Shore D Hardness	
at 75 F.	88
at 200 F.	87
at 300 F.	86
at 400 F.	84
Ultimate Compressive Strength	
after 24 hours in boiling 30% H <sub>2</sub> SO <sub>4</sub> .	22,000 psi
after 24 hours in boiling 15% NaOH.	14,600 psi
Cure Shrinkage.	2.0 to 2.5%

\* Jet-Kote X-8 manufactured by Furane Plastics, Inc.

TABLE 2—Hardness and Shrinkage Values of a Typical Furane Resin

	Shrinkage	Shore D (Inch/10 Sec.)
After 24 hr at 75 F.	1.6%	50/39
Plus 2 hr at 200 F.	1.3%	86/85
Plus 16 hr at 300 F.	2.2%	88/87

## Reinforced Furane—

(Continued From Page 26)

psi reported for epoxy and polyester laminates prepared from the same cloth. Zero or contact laminating pressures were used in preparing the samples.

As indicated in Table 3, physical properties of laminated fiberglass structures fall off markedly during the first 24 hours when immersed in hot solutions. This fact has been attributed primarily to the wicking action of the glass cloth and to simultaneous stress and chemical immersion.<sup>2</sup> In commercial practice, this marked fall-off in strength is circumvented by a resin-rich layer exposed to the solution or an organic fiber surface layer to prevent chemical solution contact with glass fibers. This must be considered in the design and manufacture of chemical equipment.

Other physical data<sup>3</sup> on furane laminating resins are given in Table 4. These laminates were prepared with No. 181 structural laminating glass cloth and cured for 60 minutes at 320 F at 25 psi.

Tests were conducted in accordance with government specifications. These physical properties are greater than those in Table 3 due to the character of the glass cloth. No. 181 style, however, does not lend itself to contact pressure laminating as might be required for the manufacture of chemical processing equipment.

### Applications

Furane resin structures can be used in the following chemical industry applications:

1. Glass cloth and organic fiber surface for metal equipment liners.
2. Surfacing reinforced layer over polyester or epoxy structures for better chemical resistance.
3. Fabricated processing equipment such as pickling tanks, ducts, stacks, etc., as required by chemical, textile and plating manufacturers.

For each of these applications, phenolics, polyesters and epoxies can be used, but furane resins have better resistance to solvents, acids and alkalies than the other systems. All these materials are attacked by strong oxidizing acids, hypo-chlorites and halogens. Furanes demonstrate good chemical resistance at higher temperatures.

Some of the shortcomings of the furane resins are dark color, poor bonds to metal and high shrinkage during cure—qualities which clearly favor the epoxies.

### References

1. A. Dunlop and F. Peters, *The Furanes*, Reinhold Publishing Corp., 1953.
2. J. Delmonte, Effect of Stress and Chemical Immersion on Epoxies, SPI Reinforced Plastics Meeting, Chicago, Ill., February, 1959.
3. J. Delmonte, J. Knudsen and L. Smith, Development of Furane Resin Glass Base Laminates, Wright Field Air Development Center Technical Report 53-56, 1953.

### DISCUSSIONS

Question by E. G. Brink, American Viscose Corporation, Marcus Hook, Pennsylvania:

Have the wetting and adhesive characteristics of the furane resins with respect to glass held back the development of high glass content reinforced furane plastics comparable to the epoxies and polyesters?

Reply by J. Delmonte:

Furane resins do not have poor wetting characteristics. As a matter of fact, their wetting qualities are good and they will penetrate readily into glass cloth and semi-porous surfaces. Their appli-

cation to glass reinforced laminates has been retarded due to their slow rate of cure, a condition now alleviated by the proprietary material given in the article.

Question by Charles M. Stanbury, Horton Steel Works Ltd., Fort Erie, Ontario, Canada:

Another speaker at the Chicago NACE Conference stated that epoxies were approximately double the cost of polyesters. Could you state roughly how furane would compare in price?

Reply by J. Delmonte:

Furane resins would be priced in the vicinity of epoxies. If their volume of application grows considerably larger, they have the possibility of reaching the cost of polyesters.

Question by Irvin Baker, Baltimore, Maryland:

What types of metal primers do you recommend for application under furane coatings for ferrous metal surfaces?

Reply by J. Delmonte:

Furane coatings tend to be brittle unless the particular variety, such as the baked furane epoxy type X-5-B is employed. For our room temperature setting materials, we recommend phenolic-rubber primers.

Question by Orest B. Draganiuk, Canadian Chemical Co., Ltd., Edmonton, Alberta, Canada:

Is any data available on corrosion resistance of either epoxy or furane resins in boiling 1 percent H<sub>2</sub>SO<sub>4</sub> with ozone added?

Reply by J. Delmonte:

We do not have specific data on the corrosion resistance of epoxies or furane in boiling solutions of sulfuric acid with ozone. However, if a thermo-setting polymer is to perform satisfactorily under these conditions, we feel that a furane resin offers the most likely prospect.

TABLE 3—Physical Properties of Furane Resin (8-Ply Laminate, No. 1500 Glass Cloth)

Flexural Strength at 75 F.	20,000 psi
Flexural Strength at 200 F.	20,000 psi
Flexural Strength at 300 F.	19,000 psi
Modulus of Elasticity in Flexure at 75 F.	1.9 x 10 <sup>6</sup> psi
Flexural Strength	
after 24 hours in 150 F water.	16,600 psi
after 1 week in 150 F water.	16,300 psi
after 24 hours in 150 F—5% Sulphuric.	16,000 psi
after 1 week in 150 F—5% Sulphuric.	11,500 psi
after 24 hours in 150 F—5% NaOH.	13,600 psi
after 1 week in 150 F—5% NaOH.	7,800 psi

TABLE 4—Physical Properties of Furane Resin Laminate\*

Flexural Strength—psi.	61,700
Modulus of Elasticity—psi.	3.3 x 10 <sup>6</sup>
Ultimate Tensile Strength—psi.	48,000
Ultimate Edge Compressive Strength—psi.	37,000
Water Absorption (Change in 24 Hours).	plus 1.1%
Resin Content.	28.1%
Tested Wet After 30 Days in Water	
Flexural Strength—psi.	42,600
Modulus of Elasticity—psi.	2.5 x 10 <sup>6</sup>
Ultimate Edge Compressive Strength—psi.	24,500
After 1 Week in	
Petroleum-Base Oil	
Change in Weight.	plus 1.4%
Flexural Strength—psi.	50,400
Isopropyl Alcohol	
Change in Weight.	plus 0.7%
Flexural Strength—psi.	49,000
Hydrocarbon Fluid (Socals No. 3)	
Change in Weight.	plus 1.1%
Flexural Strength—psi.	49,800

\* Press cure, 25 psi, No. 181 glass cloth, 14 plies.

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## TECHNICAL COMMITTEE ACTIVITIES

### **T-6F Hears Reports From 5 Task Groups At Denver Meeting**

Five task group reports were given at the October 20 meeting of Technical Unit Committee T-6F (Protective Interior Linings, Applications and Methods) held during the North Central Region Conference in Cleveland, Ohio. Twenty-seven members and guests were present. Task group reports given are listed below:

**T-6F-2 Surface Preparation:** Chairman H. T. Rudolf has appointed all committee members and has completed a comprehensive outline of work to be undertaken by the task group.

**T-6F-3 Inspection:** Chairman J. L. Barker reported on the task group's progress to date and passed out several pages of their interim report.

**T-6F-4 Safety:** Jens Nielsen has been appointed chairman. The committee has been formed and an outline of proposed work has been completed.

**T-6F-5 Application:** H. W. Foelsch, chairman of the task group, has resigned.

**T-6F-6 Ethics:** This newly activated task group will be comprised of T-6F members whose company is engaged in professional application of thin and thick film type lining materials. The task group proposes to prepare stories of interest for publication that illustrate the importance of proper specification, integrity, and intelligent inspection of applied lining materials. It is hoped that this publicity will stimulate interest in the apparent need of professional code of ethics for material suppliers and applicators.

### **T-7F Ontario Committee To Have 10th Anniversary**

The Southern Ontario Committee on Electrolysis, affiliated with NACE's T-7F Canadian Region Corrosion Coordinating Committee, will celebrate its 10th anniversary at an April meeting. New officers will be elected.

Outgoing officers are Chairman Gordon I. Russel, Interprovincial Corrosion Control Co., Ltd., Burlington, Ontario; Vice Chairman L. F. Heverly, Trans-Canada Pipe Lines Ltd., Toronto, Ontario; and Secretary-Treasurer D. W. Lemon, Sarnia Products Pipe Line, Waterloo, Ontario.

Fifty-seven members and guests attended the committee's January 26 meeting held in London, Ontario. Guest speaker was M. C. Miller, who presented a soil box demonstration. He showed how various metals in contact with the soil develop a potential and how current flows from one metal to the other. He also explained the application of cathodic protection.

\*  
7464 copies of NACE Technical Committee Reports published in CORROSION were sold in 1958.

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### **Oil Well Pump Corrosion Discussed at T-1D Meeting**

Oil well pump corrosion was discussed in a task group report given at the October 13 meeting of Unit Committee T-1D (Sour Oil Well Corrosion) held during the South Central Region Conference in Denver.

Task Group T-1D-4 (Oil Field Pumps) reported that one of its projects was to publish a tabulation of materials used in the various pump parts by each manufacturer of oil well pumps. A letter awaiting final approval will be mailed to manufacturers asking for cooperation in assembling this data on pumps and pump parts.

More than 30,000 franchised retail dealers sell motor trucks in the United States.

NACE's 17th Annual Conference and 1961 Corrosion Show will be March 13-17, 1961, at the Hotel Statler in Buffalo, New York.

### **Membership in NACE Technical Unit Committees**

A question frequently asked by guests attending meetings of Technical Unit Committees is "How may I become a member of this committee?" This question is answered in the following procedure quoted from the Technical Committee Operation Manual:

1. Any member of NACE (irrespective of place or country of residence) may become a member of a Technical Unit Committee upon fulfillment of the following requirements.

2. He should apply in writing to the Chairman of the Unit in which he desires membership, stating his interest and willingness to participate in the activities of the



Kroon



Dean

### **Kroon and Dean Are Elected T-4J Officers**

Newly elected officers for Technical Committee T-4J (Cathodic Protection of Cable Sheaths) are Harry E. Kroon, chairman, and Roy O. Dean, vice chairman.

Mr. Kroon is staff supervisor in electrolysis and cable testing for Illinois Bell Telephone Co., Chicago, Ill. He has been a NACE member since 1949.

Mr. Dean, one of the first corporate members of NACE who joined the association in 1946, is supervisor of the Corrosion Engineering Department in the Bureau of Tests and Inspect for Pacific Gas and Electric Co., Emeryville, Cal. He has a BS in electrical engineering from Tri-State College.

### **T-5C To Meet Late in May**

The executive committee of T-5C-1 (Corrosion by Cooling Waters—South Central Region) discussed a tentative agenda for a meeting to be held the latter part of May.

Topics proposed for discussion at the May meeting will include pre-treatment, fouling, velocity, alloy tubes, steel tubes, cooling tower deterioration, side stream filters and round table discussion. Business meeting will also be included.

Unit and giving his experience in the subject.

3. Approval by the Chairman of the Unit Committee of the application or by majority vote of committee members by letter ballot is necessary for appointment of an applicant to membership in a Unit Committee.

4. The Chairman of the Unit Committee will notify each applicant of his acceptance or rejection.

A directory of NACE Technical Unit Committees and Officers is published in the January, April, July and October issue of CORROSION.

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*Bituplastic No. 44*, a mastic coating of troweling consistency, serves as an excellent adhesive and is ideally suited for such uses as adhering cellular glass insulating blocks to the exterior of storage tanks. Bituplastic No. 44 also offers unusual resistance to water absorption, heat and chemical fumes. Another member of the coal tar polymer emulsion coating group, Bituplastic No. 44 is also available on request in a fibrated form, designated as Bituplastic 3S.

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# NACE NEWS



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## Southeast Region Conference Chairmen Appointed for 1960

Four general topics will be included in the technical program for the 1960 Southeast Region Conference scheduled for October 6-8 at the Dinkler-Plaza Hotel in Atlanta, Ga. The four topics on basic information and practical solutions to corrosion problems are specific applications, material selection, underground structures and coatings.



Tait



West

The program for October 8 will include a luncheon and attendance at the Georgia Tech-LSU football game.

Committee chairmen who will handle arrangements for the conference are listed below:

General Chairman and Program Chairman H. T. Rudolf of Atlantic Coatings Co., Inc., Jacksonville, Fla., Local Arrangements Chairman J. B. West of Aluminum Company of America, Atlanta, Ga., Program Arrangements Chairman E. P. Tait of Alloy Steel Products Co., Atlanta, Ga., General Finances Chairman J. L. English of Oak Ridge National Laboratories, Oak Ridge, Tenn., Local Finances Chairman R. F. Trapp of Pipeguard Corp., Atlanta, Ga., Miscellaneous Arrangements Chairman D. T. Rosselle of Southern Bell Telephone, Atlanta, Ga., and Publicity Chairman Ivy M. Parker of Plantation Pipe Line Co., Atlanta, Ga.

## Southeast Region

**Ohio Valley Section** held a joint meeting with the Louisville Electrolysis Committee on February 19. Charles C. Maddux, who is to retire from Southern Bell Telephone Company April 1, was presented a gift in appreciation for his services and cooperation through the years. He has been a member of the NACE section since its organization in 1954. He has been with Southern Bell for 41 years.

**Birmingham Section** has scheduled future meetings for May 6, September 9 and November 4.

Topic for the May 6 meeting will be "Above Ground Corrosion." Guest speaker James R. Anderson of Industrial Paint Manufacturing Company, Birmingham, will discuss economic implications of above ground corrosion to industry and the uses of organic and inorganic chemical surface coatings for corrosion control.

The 1960 Southeast Region Conference will be held Oct. 6-8 at the Dinkler-Plaza Hotel, Atlanta, Ga.

## Slide-Talk Available On Surface Preparation

A color slide presentation on metal surface preparation by sandblasting is available on loan from NACE Central Office. The talk is based on a paper by E. W. Oakes, Clemencia, Ltd., Houston, presented at the NACE Annual Conference in Chicago, 1959. The illustrations used are 35mm color slides.

Requests should be sent to Gilbert Rolak, 1061 M & M Bldg., Houston 2, Texas.

## Western Region

### Portland Section Short Course To Have Nine Guest Speakers

Nine speakers for the Portland Section Corrosion Control Short Course will cover basic training topics in sessions scheduled for April 27-29 at the Public Service Building.

Nine sessions plus demonstrations have been planned. The nine 2-hour sessions with speakers are listed below:

1. Fundamentals of Corrosion, by Mark Adams of Washington State College and William Wilson of Crown-Zellerbach, Camas, Washington.

2. Recognition of Corrosion Type, by Neally Wood of Charlton Laboratories, Portland.

3. Instrumentation, by James F. Norton of Norton Consulting Engineers, Seattle, Washington.

4. Corrosion Survey, by George Silkworth of Northwest Division, El Paso Natural Gas Co., Yakima, Washington.

5. Methods of Corrosion Control, by Paul Christiansen of Copon Company.

6. Coatings for Protective Methods, by representative of Amercoat Company.

7. Literature Sources, by Eric Bromberg, librarian for Bonneville Power Administration.

8. Cathodic Protection, by Troy R. Stilley of Good-All Electric Co., Ogallala, Neb.

9. Corrosion Prevention—Altering Environments, by C. M. Schillmoller of International Nickel Co., Inc., Los Angeles, Cal.

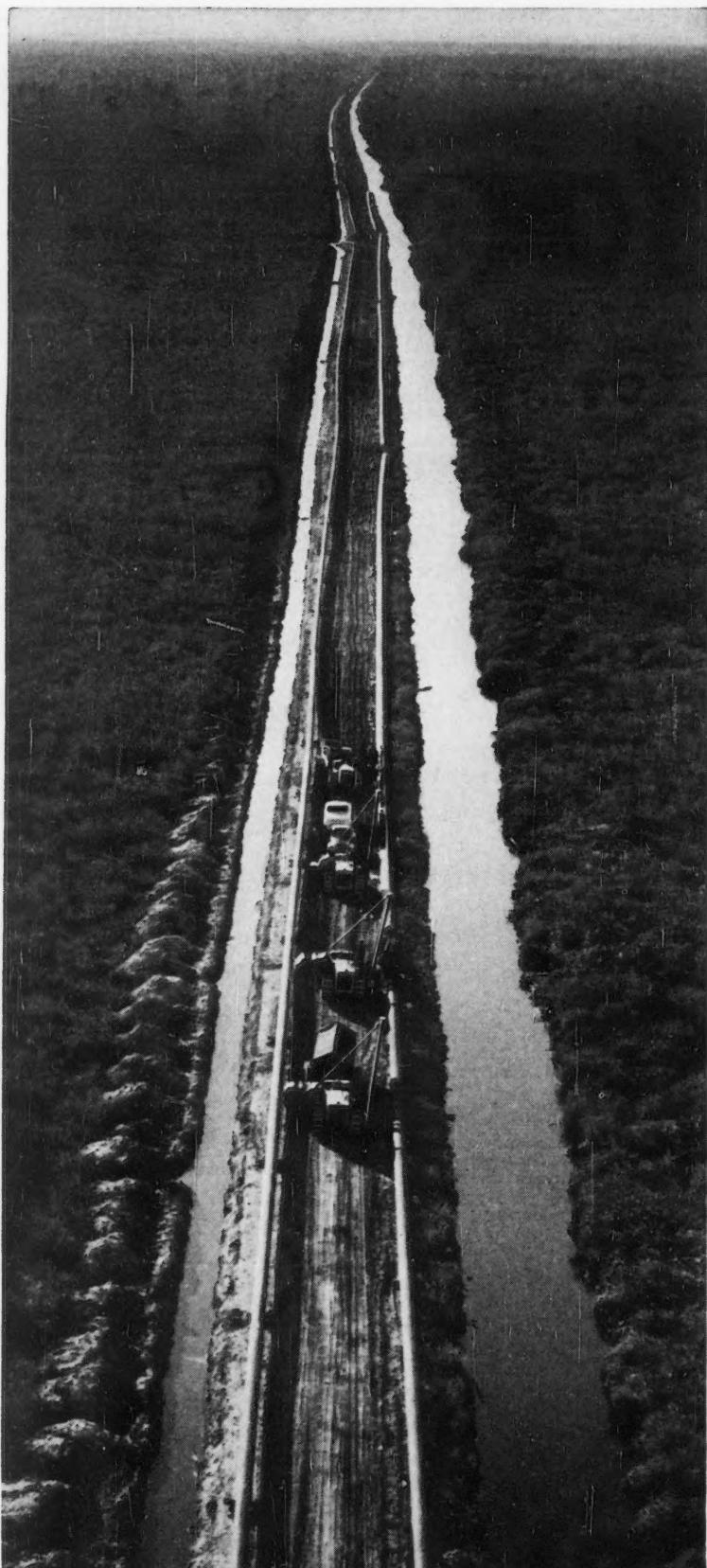
**San Francisco Bay Area Section** had a panel discussion on X-ray and gamma ray equipment as a tool for corrosion inspection at the March 8 meeting. Panel speakers were Bob Wiley of Pacific Industry X-Ray Company, Ernie Melmon of Sicular X-Ray Company and George Moller of Union Oil Company.

**Portland Section** scheduled a showing of the film "Corrosion in Action" for the March 10 meeting. Presentation was made by William Efaw.

Corrosion of underground installations was discussed by Neally Wood of Charlton Laboratories at the February 11 meeting.

Western Region's 1960 Conference will be Oct. 6-7 at the Sheraton Palace Hotel in San Francisco, Cal.

Certificates for past chairmen of regions and sections, measuring 9 x 12 inches, are available from the Central Office at \$7.50 each.



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Plastic Engineering & Sales Corp.

**Houston, Texas**  
Cathodic Protection Service

**Jackson, Michigan**  
Utility & Industrial Supply Co.

**Kansas City, Missouri**  
H. J. Hodes Co.

**Kansas City, Missouri**  
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**Long Beach, Calif.**  
Barnes & Deane

**Memphis, Tenn.**  
General Pipe & Supply Co.

**Minneapolis, Minn.**  
Simcoe Equipment Co.

**New Orleans, Louisiana**  
L. F. Gaubert & Co.

**Philadelphia, Pa.**  
Harold N. Davis Co.

**Plainfield, New Jersey**  
Stuart Steel Protection Corp.

**San Francisco, Calif.**  
Incandescent Supply Co.

**San Francisco, Calif.**  
Phillips & Edwards Electric Co.

**Seattle, Washington**  
Farwest Corrosion Control Corp.

**Seattle, Washington**  
Pacific Water Works Supply Co.

**St. Louis, Missouri**  
Shutt Process Equipment Co.

**Tulsa, Oklahoma**  
Midwestern Pipe Line Products Co.

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Experienced in modern  
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## NACE Member Visits Israel As UN Representative

NACE member Francis W. Ringer, a consulting corrosion engineer of Narberth, Pa., recently spent about two months in Israel as a representative of the United Nations to discuss water planning problems.

While in Israel, Mr. Ringer led a round table conference on cathodic protection for the local corrosion group, headed by another NACE member, D. Spector of Tel Aviv.

## South Central Region

**Shreveport Section** had 56 members attending the March 1 meeting. Guest speaker was Dorsey Fincher of Tide-water Oil Company, Houston, Texas, whose topic was "Design of Equipment for High Pressure Well Completions."

**Houston Section** had a panel discussion on the subject "Corrosion—A Future?" for its March 8 meeting, designated as students' night. The panel discussed the future of corrosion control as a profession and the challenging problems ahead. Students were invited from the University of Houston, Texas A & M College, University of Texas, Rice Institute and Lamar Tech.

Panel members were Russell A. Branion, Humble Pipe Line Co., J. Curtis Lloyd, Tennessee Gas Transmission Co., Walter F. Rogers, Gulf Oil Corp., and William A. Wood, Jr., Carbofine Company. Panel moderator was M. A. Riordan, Rio Engineering Company.

**Greater Baton Rouge Area Section** had a slide presentation on sandblasting and a discussion on the economics of each class of sandblasting for its February 22 meeting. Woody Gilpin of K-Way Equipment Company made the presentation.

**East Texas Section** heard B. Wooley of Wooley Tool Co., Kilgore, speak on casing back-off at the February 23 meeting.

**Central Oklahoma Section** had two guest speakers for the February 8 meeting. They were John Barry and Louis Perret of the Carlton Products Company. Their topic was development of thermoplastics for oil field pipe.

**Tulsa Section** scheduled a talk on protective coatings for the petroleum industry for the March 28 meeting. Guest speaker was Jack P. Barrett of the Pan American Research Center.

The 1960 South Central Region Conference will be held October 25-28 at the Mayo Hotel, Tulsa, Okla.

News deadline for CORROSION is the 10th of each month.

## DEATHS

**F. E. Wilson**, NACE corporate member representing Clemtex, Inc., Houston, died after a heart attack February 20. He was co-owner of the company.



## SECTION CALENDAR

### April

- 4 North Texas Section.
- 5 Shreveport Section. Capt. Shreve Hotel. Review of 1960 NACE Conference.
- 7 West Kansas Section.
- 7 Pittsburgh Section. Ladies' Night.
- 12 San Francisco Bay Area Section.
- 19 Chicago Section. Drainage Problems.
- 19 Baltimore-Washington Section. Corrosion Preventive Measures Necessary in Design and Construction of an Underground Pipeline, by E. C. Hinie and R. E. Barrans.
- 21 Southwestern Ohio Section. Evaluation of Coatings for Protection of Steel at Elevated Temperature, by Edward Norris of Metcut Research Assoc., Inc., to be held at Connersville, Ind.
- 25 Tulsa Section.
- 26 Panhandle Section.
- 26 Detroit Section. Meeting concurrent with ASTM. Speaker to be Frank LaQue of International Nickel Co., Inc.
- 28 Edmonton Section.
- 28 Sabine-Neches Section.

### May

- 2 North Texas Section.
- 3 Shreveport Section. Capt. Shreve Hotel. Cathodic Protection—Operation and Maintenance.
- 5 Schenectady-Albany-Troy Section. Oxidation of Iron Chromium Alloys, by C. E. Birchenall.
- 5 West Kansas Section.
- 5 Pittsburgh Section. Plant tour of U. S. Steel's National Tube Division, McKeesport, Pa.
- 6 Birmingham Section. Above Ground Corrosion, by T. G. Humphreys, Jr., Alabama Gas Corp.
- 9 Greater St. Louis Section. Newness of Thin Film Linings, by W. P. Cathecart, Tank Lining Corp., Pittsburgh, Pa.
- 10 San Francisco Bay Area Section.
- 10 Permian Basin Section.
- 11 Greater Boston Section. Election of officers. Analysis of Furane and Other Plastics in Corrosion Control, by John Delmonte, Furane Plastics, Los Angeles.
- 17 Chicago Section. Cost Evaluation.
- 18 Los Angeles Section.
- 19 Detroit Section. Forum discussion and exhibit on non-destructive testing for corrosion control.
- 23 Tulsa Section.
- 26 Edmonton Section.
- 26 Sabine-Neches Section.
- 31 Panhandle Section.
- 31 Philadelphia Section. Metallizing—State of the Art.
- 31 Southwestern Ohio Section. Corrosion Prevention by Bituminous Coatings, by Donald Burke, Philip Carey Mfg. Co. To be held in Cincinnati.

## St. Louis Section Gives Topics for 1960 Meetings

Guest speakers and topics for four future meetings have been set by the Greater St. Louis Section. Dates are May 9, September 12, October 10 and November 14.

On May 9, W. P. Cathcart of Tank Lining Corp., Pittsburgh, Pa., will speak on "Newness of Thin Film Linings." Guest speaker for the September 12 meeting will be John Bobbin of Branson Instruments, who will talk on "Non-Destructive Test Methods." Topic for October 10 will be "Alloys in the Process Industries," given by John Schley of the Haynes-Stellite Company. C. R. Martinson of Monsanto Chemical Co., St. Louis, will speak on "Roofing Materials for Chemical Plants" at the November 14 program.

Southwestern Ohio Section heard Har-

old Boerger of the Hilton Davis Company speak on "Materials of Construction in the Chemical Process Industries" at the February 23 meeting.

Kansas City Section will have four speakers for the April 11 meeting to be held at the Wish Bone Restaurant. R. H. Goodnight of Cook Paint Company will discuss coal tar epoxies. R. English of Minnesota Mining & Manufacturing will talk on plastic tapes. E. W. Roberts of Dearborn Chemical Company will speak on asphalts and waxes. J. R. Robinson of Koppers Company, Inc., will discuss coal tar coatings.

At the February 8 meeting, Daniel R. Werner of American Telephone and Telegraph Company spoke on construction of deep anode ground beds.

Detroit Section had R. Gackenbach of American Cyanamid Co., New York City, as guest speaker for the March 24 meeting. His topic was "Paint, a New Approach to an Older Art."

The 1960 North Central Region Conference will be held Oct. 19-20 at the Schroeder Hotel in Milwaukee, Wis.

## POSITIONS WANTED AND AVAILABLE

Active and Junior NACE members and companies seeking salaried employees may run without charge two consecutive advertisements annually under this heading, not over 35 words set in 8 point type. Advertisements to other specifications will be charged for at \$10 a column inch.

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### 1960

Oct. 6-7—10th Annual Western Region Conference, Sheraton-Palace Hotel, San Francisco, Cal.

Oct. 6-8—Southeast Region Conference, Dinkler-Plaza Hotel, Atlanta, Ga.

Oct. 11-14—Northeast Region Conference, Prichard Hotel, Huntington, W. Va.

Oct. 19-20—North Central Region Conference, Schroeder Hotel, Milwaukee.

Oct. 25-28—South Central Region Conference, Mayo Hotel, Tulsa.

### 1961

March 13-17—17th Annual Conference and 1961 Corrosion Show, Buffalo, N. Y., Hotel Statler.

Oct. 4-6—Western Region Conference, Hotel Multnomah, Portland, Oregon.

Oct. 9-11—North Central Region Conference, St. Louis, Chase Park Plaza Hotel.

Oct. 24-27—South Central Region Conference, Houston, Shamrock Hotel.

Oct. 30-Nov. 2—Northeast Region Conference, New York City, Hotel Statler.

Southeast Region Conference, Miami, Fla., in conjunction with Miami Section's short course.

### 1962

March 19-23—18th Annual Conference and 1962 Corrosion Show, Kansas City, Municipal Auditorium.

October 1-4—Northeast Region Conference, Hotel Sheraton Ten Eyck, Albany, N.Y.

October 9-11—North Central Region Conference.

October 16-19—South Central Region Conference, Hilton Hotel, San Antonio, Texas.

Southeast Region Conference, Birmingham, Ala.

Western Region Conference, San Diego, Cal.

### SHORT COURSES

#### 1960

April 27-29—Portland Section Corrosion Control Short Course.

May 11-13—Niagara Frontier Section Symposium on Organic Coatings. Hotel Niagara, Niagara Falls, N. Y.

June 1-3—5th Annual Appalachian Underground Corrosion Short Course, West Virginia University, Morgantown, W. Va.

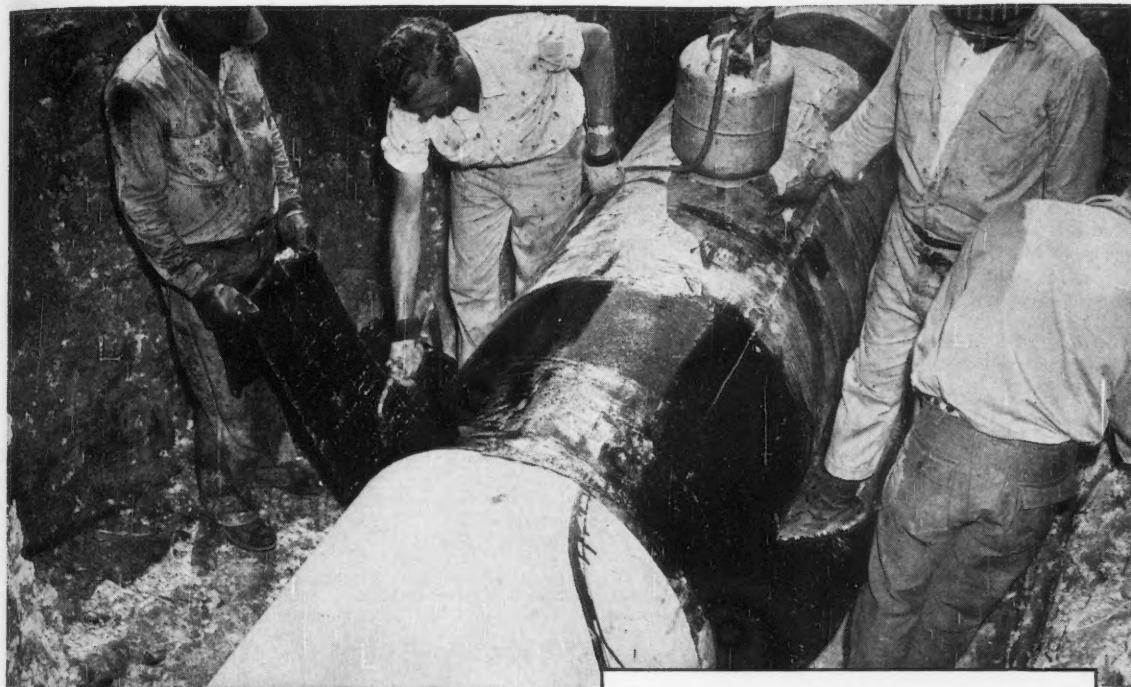
September 28-30—Central Oklahoma Section 1960 Corrosion Control Short Course, at University of Oklahoma, Norman.

October 3-5—Corrosion Control Short Course, sponsored by Western Region and University of California, San Francisco.

In 1958, there were over 11 million privately and publicly owned trucks on U. S. streets and highways, representing 17 percent of the total U. S. motor vehicle registrations.

News intended for publication in CORROSION should be in Houston no later than the 10th of the month preceding month of publication.

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## Northeast Region

### May 11-13 Organic Coatings Symposium Program Completed

Final program arrangements have been completed for the May 11-13 Organic Coatings Symposium sponsored by the Niagara Frontier Section. Theme of the symposium will be proper use of coatings for protection of ferrous surfaces.

The three-day program will be held at the Hotel Niagara. Schedule for the symposium is given below:

**May 11, morning:** Discussions of surface preparation methods to include sandblasting (both open and confined), electric power tools and chemical cleaning.

### Letters to the Editor

19 Manor Court  
Grange Road  
Cambridge, England  
February 8, 1960

On pages 120-123 of your January issue, you reproduced the Translations of Foreign Corrosion terms from DIN 50900, and say "credit for this work is due to U. R. Evans . . . who compiled the English version." This attributes to me a task which was really performed by Dr. Kutzelnigg of Nurnberg.

What happened was that my friend, Dr. Schikorr, kindly sent me a printer's proof of the DIN and invited comments. I looked through the list and suggested slight improvements of the English equivalents in perhaps a dozen cases; in some instances, I gave more than one form of expression and asked him to choose which he liked; a certain amount of correspondence followed. I think, in connection with paraphrasing in cases where there appeared to be no simple equivalent for the single German words. I rather imagined that other English speaking corrosion scientists would be offering their suggestions, and I note that my friend and former colleague Dr. Mears has added a valuable appendix.

The acknowledgement in the DIN states that Dr. Evans *besorgte* the English list, and this appears to me a fair description of the situation; it does not mean that I "compiled" the list. The word *besorgen* has many uses; a familiar one occurs when someone is asked *die Kinder ein paar Stunden zu besorgen* (to look after the children for an hour or two); this conveys responsibility for minor correction but introduces no suggestion of parentage.

signed,  
Ulick R. Evans

## Canadian Region

Edmonton Section had G. S. Rice, president of Internal Pipeline Maintenance Company (Texas), speak on "Internal, In-Place Cleaning and Coating of Pipe Lines" at the February 25 meeting.

**May 11, afternoon:** Demonstrations of all methods discussed.

**May 12, morning:** Discussions on methods of application to include brush, pressure spray, hot spray, airless spray, pressure rollers and conventional rollers.

**May 12, afternoon:** Demonstrations of all application methods discussed.

**May 13, morning:** Discussions on generic types of coatings to include vinyls, epoxies, urethanes, esters, chlorinated rubber, chlorofluorinated polyethylene alkyls and phenolics.

**May 13, afternoon:** Round table discussion on problems involving industrial painting. Questions should be submitted to Paul Gegner, Round Table Chairman, Columbia-Southern Chemical Corp., Barberio, Ohio.

Luncheon speakers will discuss methods of organizing effective painting departments and easy methods for recording costs of paint jobs.

Program chairman for the symposium is Raymond E. Fonner of Rowe Products Inc., Niagara Falls, N.Y.

**Pittsburgh Section** will have a ladies' night dinner meeting April 7 at which new section officers will be elected. The meeting will be held at the Pittsburgh-Hilton Hotel. A tour of the new hotel will be included in the evening's activities.

F. L. Whitney, Jr., of Monsanto Chemical Company spoke on "Design for Corrosion Prevention in the Process Industry" at the March 3 meeting.

**Genesee Valley Section** has scheduled John Hoffer, metallurgist with Eastman Kodak Company, as guest speaker for the April 26 meeting to be held at the Rochester German Club.

The section's annual business meeting is set for May 24.

Herman Wissenberg of Union Carbide's Metal Research Laboratories, Niagara Falls, N. Y., discussed improved corrosion resistance of titanium-noble metal alloys at the February 23 meeting. Speaker for the March 29 meeting was Edward Robb, president of the Bisonite Company, who spoke on plastic coatings for above ground use.

**Schenectady-Albany-Troy Section** will hold its two final meetings of the 1959-60 year on April 7 and May 5.

D. Groot, manager of corrosion and coolant chemistry at Knolls Atomic Power Laboratory, General Electric Co., will present a paper on "Extrapolation of Corrosion Data" for the April 7 meeting.

At the May 5 meeting, C. E. Birchall will present a paper on "Oxidation of Iron Chromium Alloys."

At the March 3 meeting, guest speaker N. D. Greene of Rensselaer Polytechnic Institute discussed revolution of corrosion theory.

**Metropolitan New York Section** officers for 1960 are Chairman B. C. Lattin, Vice Chairman S. N. Palica and Secretary-Treasurer R. E. Gackenbach.

**Greater Boston Section** heard Frank L. LaQue of International Nickel Co., Inc., speak on "What We Know and Don't Know About Corrosion" at the section's March 9 meeting.



## KENTUCKY PIPELINE CONSTRUCTION JOB SAVES TIME AND MONEY WITH ROSKOTE COLD-APPLIED MASTICS

Petroleum Exploration, Inc., specified the application of ROSKOTE PIPE MASTICS, by the ROSKOTER method in the construction of its new transmission pipeline from Manchester to London, Kentucky.

**The results —**

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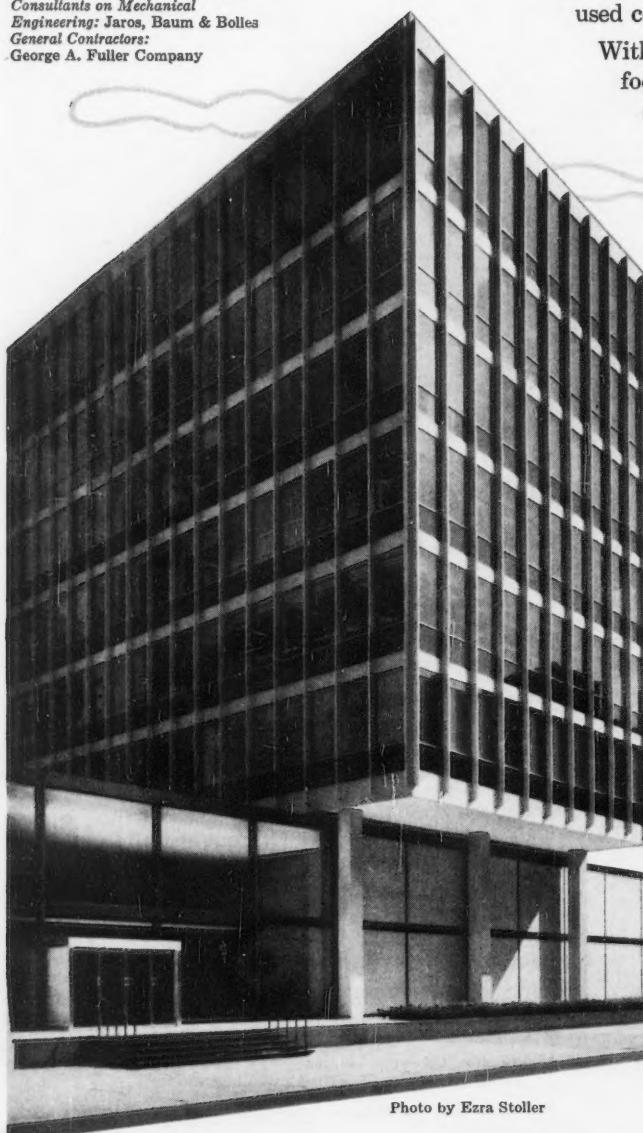


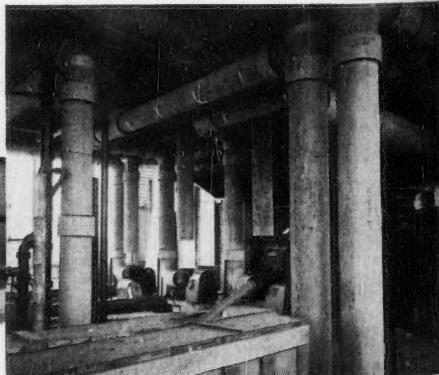
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**1960  
CORROSION  
SHOW**

## Over 2100 Registrants Attend Dallas Conference

Over 2100 total registrations were counted for the 16th Annual NACE Conference held in Dallas March 14-18. Although not a record attendance, the conference was acclaimed by many registrants as one of the best held by NACE in several years.

The smooth operation of all phases of the program reflected the advance planning and hard work of the local arrangements committee and of General Conference Chairman J. Gordon Meek, Metal Goods Corp. Mr. Meek, members committees and everyone concerned with the operation of the conference paid tribute to the foundation laid by J. C. Spalding, Jr., general conference

chairman, who died suddenly in the fall of 1959.

Mrs. J. C. Spalding, Jr. and Mrs. R. A. McCarthy, both of Dallas were in charge of the program for the unexpectedly large number of ladies who attended.

Highlight of the social events was the annual banquet at which the annual NACE awards were made and the 1959 Young Author recognized. L. L. White-neck, Los Angeles Harbor Dept., immediate past president and chairman of the NACE Awards committee, presented the Willis Rodney Whitney Award to Morris Cohen, National Research Council, Ottawa. He was the first Canadian to be so recognized by NACE.

J. C. Hudson, who received the Frank Newman Speller Award, made a short address of thanks in which he gave credit for much of his success to his co-workers in England and acknowledged the influence of Dr. Speller, first recipient of the award, and after whom it is named. Dr. Speller, as is his custom, was present at the banquet and fully enjoying the proceedings.

N. D. Greene, Jr., Rensselaer Polytechnic Institute, Troy, N. Y. received the 1959 Young Author Award from Norman Hackerman, University of Texas, Austin, chairman of the Education Committee.

The entertainment which followed the banquet, awards and transfer of authority to the new officers of the association was enthusiastically enjoyed by those present. Attendance at the banquet was in excess of 1000, the largest number to be present at an NACE annual banquet.

The reports of standing committees and special committees were heard at the annual business meeting Wednesday morning at the auditorium. The actions of the board during the preceding year were ratified.

### Subscription Price Is Raised For Some Foreign Countries

An increase in the subscription price for CORROSION for countries outside the North American Continent was authorized by the NACE Publication Committee at its meetings in Dallas in March. The new rate will be \$15.



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Certificates of membership in the National Association of Corrosion Engineers will be issued on request at \$2 each, remittance in advance. The certificates, which measure 5½ x 8½ inches, are signed by the president and executive secretary of the association.

### CERTIFICATES FOR PAST CHAIRMEN OF REGIONS AND SECTIONS

Certificates measuring 9 x 12 inches in size, prepared from an engraved plate, are available for issuance to regional and sectional chairmen. They will be supplied on request of the region or section at \$7.50 each to be paid by the region or section, the cost to be classified as a non-reimbursable expenditure.

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## **Wet SO<sub>2</sub> line now in 4th year ...proves superiority of Ni-O-Nel alloy**

*... handles severely corrosive hot acids and oxidizing chemicals, too*

That return gas line running down between the Jenssen acid towers shown above has been working there since 1955. It is at Scott Paper Company's Detroit plant.

The line is 16 inches in diameter and 77 feet long. Yet it was put up with no intermediate supports. It needed none, because of the extra strength and rigidity that Ni-O-Nel nickel-chromium-iron alloy provides.

This line carries wet SO<sub>2</sub> between the towers handling weak and strong acids. It's an outstanding replacement where a previous pipe failed.

Periodic inspections reveal no leaks or need of maintenance.

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Ni-O-Nel alloy has demonstrated in both laboratory and industry remarkable resistance to hot acids and oxidizing chemicals.

Chemical processors find it useful in handling hot sulfuric acid pickling baths, phosphoric acid up to 280° F., boiling organic acids such as acetic, phthalic and maleic, and nitric acid solutions at practically all temperatures and concentrations.

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# **N-I-O-N-E-L.**



**TYPICAL OF THE AUDIENCES** at Educational Lectures was this one (top) to hear T. P. May, The International Nickel Co., Inc., Wrightsville Beach, N. C. speak on galvanic corrosion. (Middle) This panel at the Pipe Line and Underground Corrosion Round Table Session consisted of (left to right) Charles L. Woody, United Gas Corp., Houston; Lyle R. Sheppard, Shell Pipe Line Corp., Houston; Lance F. Heverly, Trans-Canada Pipe Line Co., Toronto; and Art Erickson, People's Natural Gas Co., Pittsburgh. (Bottom) T. J. Maitland, American Telephone and Telegraph Co., New York, presides at a meeting of T-4 committee.

## Current Research Problems To Be Published As Series

Current corrosion research problems and some keys to their probable solutions will be considered in a series of articles to be submitted for publication in CORROSION. The Research Subcommittee, a unit in the NACE technical com-

mittee organization, has adopted a scope which calls for it to encourage corrosion research.

Al Roebuck, The Western Co., Fort Worth, chairman said the committee's aims were to seek practical solutions for corrosion problems and compile estimates of what areas of corrosion research are likely to produce the best results.

## Technical Meetings Are Well Attended

The common characteristic of symposia sessions during the Dallas Conference in March was good attendance. In some instances, this took the form of standing room only. The facilities available at the Dallas Memorial Auditorium were large enough to accommodate the large audiences at all sessions of the three concurrent symposia.

As has been experienced in the past, with a few exceptions related to improper scheduling, the three educational lectures were presented to overflowing audiences. The popularity of these lectures increases from year to year and it is noteworthy that prominent among those attending may be seen corrosion engineers of many years' experience.

Good attendance carried over to the Friday morning round table sessions also. At both the Pipeline and Underground and General Corrosion Problems sessions large audiences participated. The Marine Corrosion Symposium, also given on Friday morning, was well attended.

Hundreds of copies of papers were ordered from NACE on the forms provided and many registrants took forms with them to be filled in and sent in later by mail. Two other schedules of papers, those whose authors indicated copies would be available in advance of publication and those concerning which NACE had no specific information, also were provided and widely used.

Stacks of advance copies of papers provided by authors were placed on a table near the NACE booth in the Corrosion Show. These stacks melted away rapidly as registrants picked them up.

As usual, the NACE booth was the focus of considerable activity as registrants came by to get information, order books or make application for membership. Three members of NACE headquarters staff manned the booth during the show.

The 1960 Southeast Region Conference will be held Oct. 6-8 at the Dinkler Plaza Hotel, Atlanta, Ga.

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Cominco Brand Ribbon Anodes are designed for low cost cathodic protection of piers, ship hulls, ballast tanks, heat exchangers, pipe lines and many other applications.

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 Anodes have a full length bonded insert to permit complete use of the anode metal. For ease of installation and low replacement cost, Ribbon Anodes are bolted to studs welded to the structure being protected.

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### CONFERENCE SNAPSHOTS

(1) Between technical meetings, informal groups relaxed in the lobbies as this quartet composed of Donald D. Thetford, Humble Pipe Line, Cisco, Texas, James W. Sherrow, Humble Pipe Line, Longview, Texas, Kirby E. Chancey, Humble Pipe Line, Houston, Texas, and Gene E. Smith, Lone Star Steel Co., Lone Star, Texas. (2) Ed Muehlhouse, who was local committeeman in charge of housing, found time to relax. (3) A square dance exhibition was part of the Fellowship Hour. (4) After the first educational lecture session adjourned, discussions continued on the floor in informal groups such as these shown. (5) Among the many foreigners who attended the Dallas conference were these two gentlemen from Italy: Vacchelli Pierluigi and Alberto Grandi, both from Milano, shown talking with Mrs. Nicole Atlas, NACE central office staff member in the Abstract Office. (6) Three of the exhibitors are shown chatting during the Corrosion Show. (7) Comparing notes on galvanic anodes after the meeting of Technical Committee T-2A are John W. Caldwell of Bunker Hill, San Francisco, Cal., T. J. Lennox, Jr., American Smelting and Refining Co., South Plainfield, N. J., Gene E. Smith, Lone Star Steel Co., Lone Star, Texas, and S. Tudor, New York Naval Shipyard, Brooklyn, N. Y. (8) Two exhibitors take a coffee break between crowds at the Corrosion Show booths. (9) The NACE Research Committee held a dinner meeting at the Statler-Hilton Hotel on Monday, March 14. They are E. B. Backenstos, Socony Mobil Oil Co., Paulsboro, N. J., S. K. Coburn, U. S. Steel, Pittsburgh, Pa., T. J. Maitland, American Telegraph and Telephone, New York, N. Y., Chairman, A. H. Roebuck, Western Co., Fort Worth, Texas, and J. A. Caldwell, Humble Oil & Refining, Houston, Texas. (10) Group of the Corrosion Show delegates from the Latin American countries of the Fellowship Hour. (11) Frans Vander Henst, NACE Technical Committee Secretary at Houston, and E. J. Simmons, conference properties chairman, make last minute arrangements for a rostrum and slide projector. At left is Fred M. Reinhart of the National Bureau of Standards, Washington, D. C. At right is C. L. Mercer of Southwestern Bell Telephone, Houston. (12) Crowds gathered at the Corrosion Show booths Tuesday afternoon.

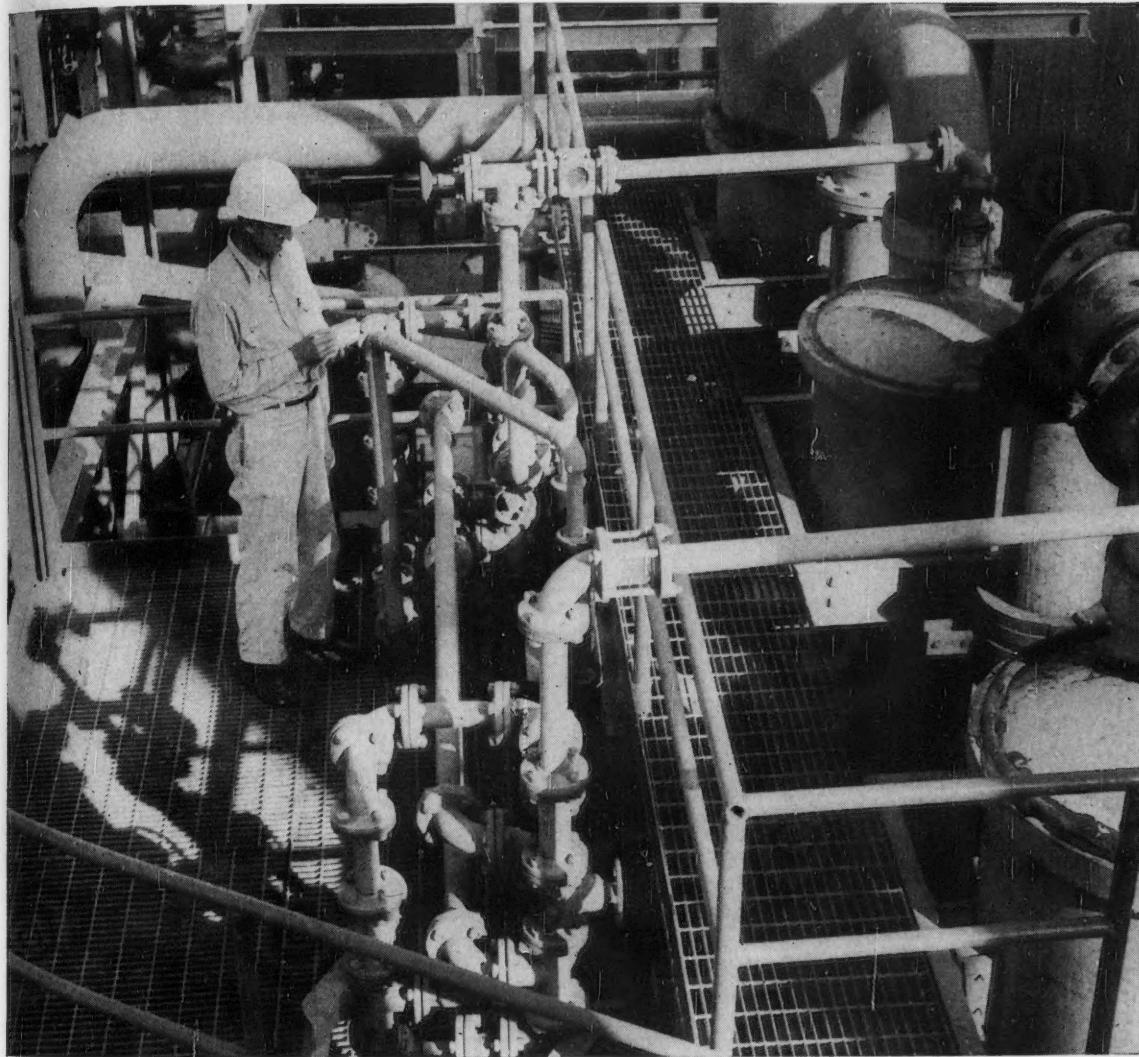
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## SARAN LINED PIPE



### After 16,000,000 Gallons of Sulphuric Acid ... No Corrosion in **SARAN LINED PIPE**

A vital round-the-clock stream of highly corrosive  $H_2SO_4$  . . . to be carried by a piping complex that *must not* shut down for any reason . . . calls for the ultimate in corrosion resistance. Thanks to Saran Lined Pipe, the installation shown above has carried over 16,000,000 gallons of acid without a shutdown, or trace of corrosion, since construction a year ago.

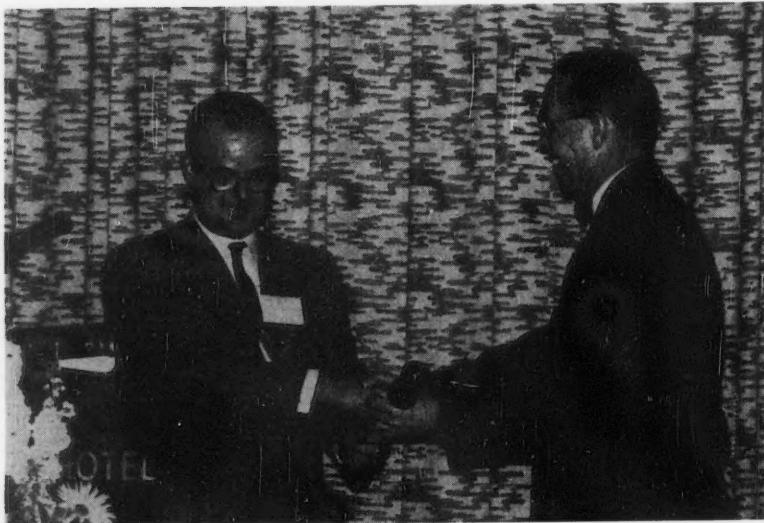
At the Gramercy, Louisiana, plant of Kaiser Aluminum & Chemical Corporation, thirty gallons per minute of sulphuric acid are pumped through this three-inch Saran Lined Pipe

into vertical tanks where newly generated chlorine gas is dried. Failure in these lines would shut down the entire caustic-chlorine plant, but the corrosion resistance of the Saran Lined Pipe permits uninterrupted operation, month after month.

Saran Lined Pipe, fittings, valves and pumps are available for systems operating from vacuum to 300 p.s.i., and from below zero to 200° F. They can easily be cut, fitted and modified in the field. For information write, Saran Lined Pipe Company, 2415 Burdette Ave., Ferndale, Mich., Dept. 2285 AU4.

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HUGH P. GODARD, outgoing NACE president transfers the gavel of authority to George E. Best incoming president at the annual NACE banquet at Dallas. Left, top, Morris Cohen (left) receives from L. L. Whiteman, the certificate exemplifying the Whitney Award; middle, J. C. Hudson receives certificate exemplifying the Speller Award; bottom, N. D. Greene, Jr. (left) receives from Norman Hackerman the 1959 Young Author Award.

### Godard Becomes Chairman Of ISCC Subcommittee

H. P. Godard, Aluminium Laboratories, Ltd., Kingston, Ont., immediate past president of the National Association of Corrosion Engineers is new chairman of the foreign correspondents' subcommittee of the Inter Society Corrosion Committee. The committee, an affiliate of NACE, held its annual meeting at Dallas during the 16th conference with 16 representatives of member organization present.

### Technical Papers Solicited For 1961 Buffalo Conference

D. K. Priest, Pfaudler-Permutit, Inc., Rochester, N. Y. chairman of the Technical Program Committee for the NACE 17th Annual Conference at Buffalo in March 1961 is seeking papers suitable for presentation at this meeting. Any who have papers they consider suitable are invited to write to him.



Portland Section's Corrosion Control Short Course will be held April 27-29.

## "Corrosion" to Have More Comprehensive Technical Coverage

Increased efforts to make *Corrosion's* presentation of corrosion control information comprehensive on a world-wide basis are going to be made in the future. Several actions by the NACE publication committee indicate this to be the essential aim for the NACE monthly magazine for the immediate future.

Actions taken supporting this intention included increased emphasis on applicatory articles in the Technical Topics Section to improve their timeliness and readability, better presentation for the articles, some minor internal rearrangements intended to build readership; more news about activities abroad on corrosion control and a list of advertised products for the convenience of readers seeking this kind of information.

The committee also recommended to the NACE board, which concurred, that an associate editor would be needed to help in publishing the applicatory articles and news portion of the magazine. The committee also sees an increased number of pages in the technical section concurrent with a sharper look at the quality of the material recommended for publication there.

A subscription schedule designed to put subscribers on a calendar year basis also was approved. This is aimed at putting subscribers in a better position to fully use the annual alphabetical index and encourage the retention of copies in libraries.

### New Membership Directory To Be Published in 1961

Publication of a membership directory of NACE in 1961 was authorized by the association's board at a meeting Sunday, March 13, in the Adolphus Hotel, Dallas. The directory will consist of an alphabetical and a geographical list of members and will be distributed free of charge to all members.

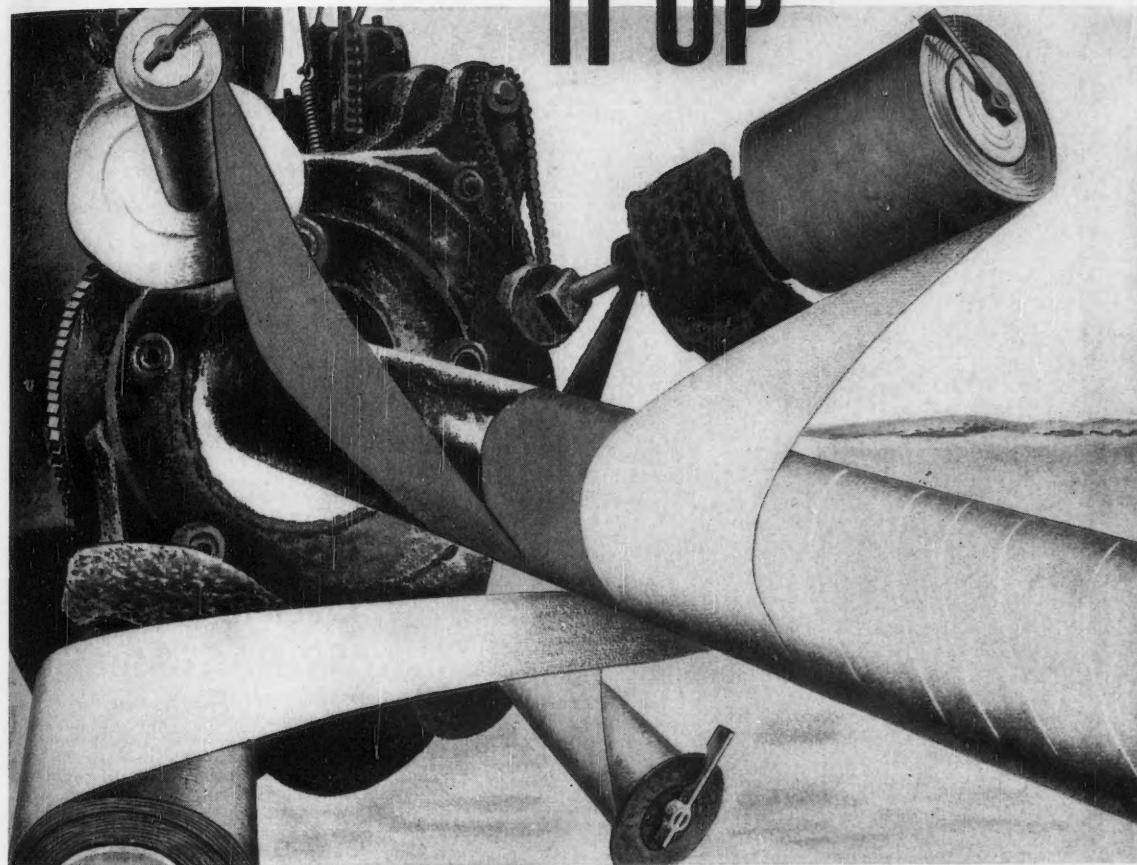
The board also authorized the executive secretary to set a schedule of firm prices for reprints of technical material published by the association.



CONFERRING ON 1961 TECHNICAL PROGRAM matters are, D. K. Priest, Technical program committee chairman (far right) and, left to right, front row, J. F. Vanderhent, NACE Central Office, Houston; David Roller, Magna Products Co., Santa Fe Springs, Cal.; second row, N. D. Casdorph, Union Carbide Chemicals Co., Port Lavaca, Texas; E. H. Phelps, United States Steel Corp., Monroeville, Pa.; C. L. Goodwin, Portland Pipeline Corp., Portland, Me.; J. J. Halbig, Sr., Armco Steel Corp., Middletown, Ohio; J. M. Bielosky, Koppers Co., Inc., Verona, Pa.; F. B. Hamel, Standard Oil Company, Cleveland; third row, S. K. Coburn, United States Steel Corp., Monroeville, Pa.; W. L. Miller, Lynbrook, N. Y.; Sidney Tudor, New York Naval Shipyard, Brooklyn; H. C. Boone, Peoples Gas, Light & Coke Co., Chicago, Ill.; D. L. Burns, Gulf Oil Co., Port Arthur, Texas; L. W. Gleekman, Wyandotte Chemicals Corp., Wyandotte, Mich.; W. A. Szymanski, Hooker Electrochemical Co., Niagara Falls, New York; and O. H. Fenner, Monsanto Chemical Co., St. Louis.

A PIPE LINE'S "HOLIDAYS" ARE FEW AND FAR BETWEEN WHEN

# FIBERGLAS WRAPS IT UP



## NEW FIBERGLAS\* OUTER WRAP JOINS INNER WRAP IN CORROSION PROTECTION

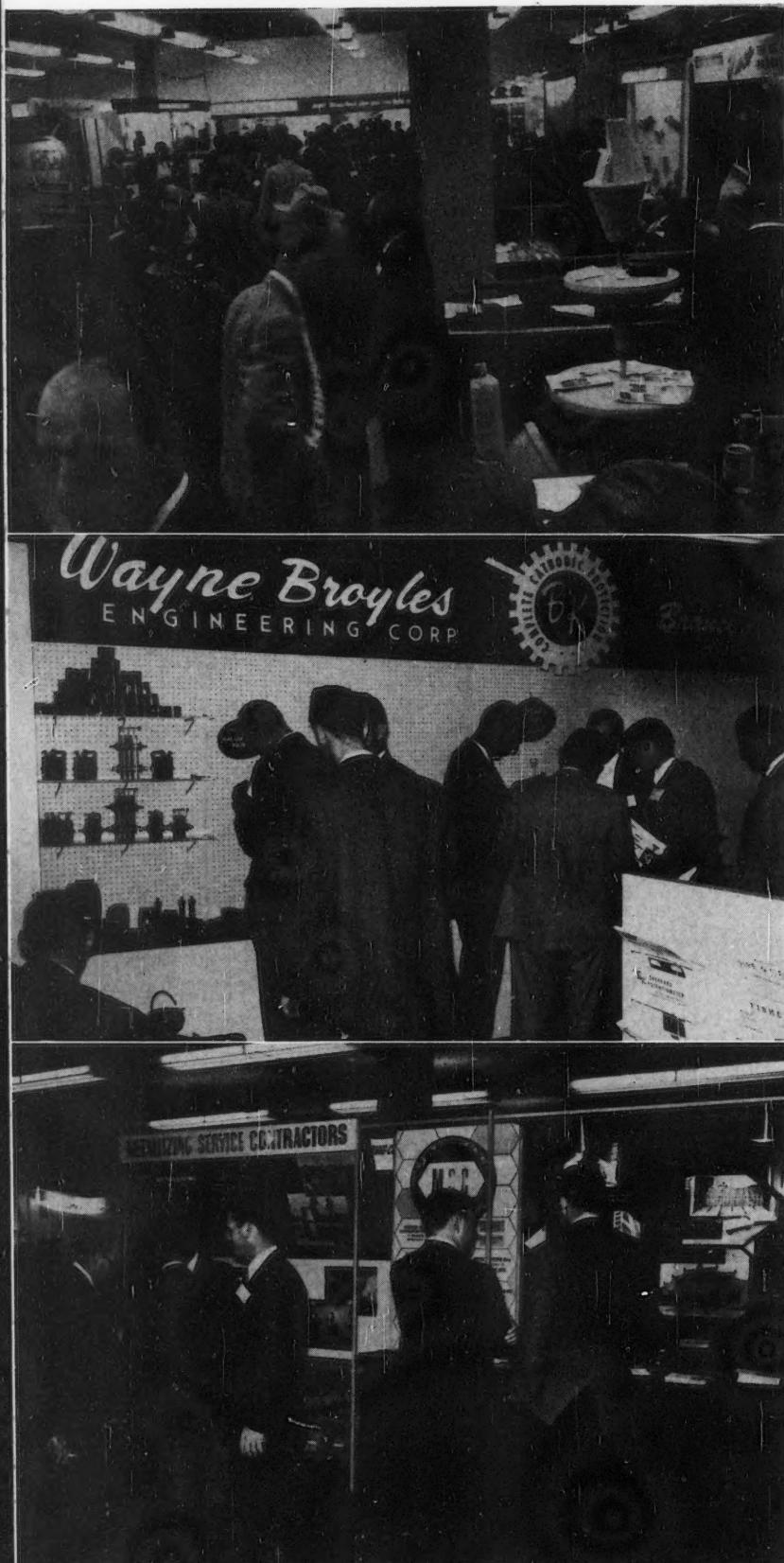
Effective corrosion protection for underground pipe lines depends on the permanence, toughness and uniformity of pipe coatings. Enamel alone can't do a complete job. But when the right kind of reinforcement—Owens-Corning Fiberglas—is added to the enamel, the physical properties of the coating are improved and maximum protection made possible. There are fewer "holidays"—imperfections in the wrapping which lead to corrosion—and fewer breaks per mile. High-strength inorganic, non-cellular glass fibers reinforce the enamel so the coating stays in place to combat the corrosive forces continually at work underground.

Fiberglas Inner Wrap has compiled an outstanding record in reinforcing enamel. Now, as a companion product

to its Inner Wrap, Owens-Corning Fiberglas introduces its new Outer Wrap. It's designed to act as an exterior protection for underground pipe lines from stresses of lowering and backfilling and from later soil movement. Superior Inner and Outer Wrap for underground pipe lines is another successful application of Fiberglas in industry. Find out about the advantages of Owens-Corning Anti-Corrosion products. Phone the Owens-Corning Fiberglas office nearest you. Or Owens-Corning Fiberglas Corp., Dept. 53D-29, Toledo 1, Ohio.

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\*T.M. (Reg. U.S. Pat. Off.) O-C.F. Corp.



## 1960 Corrosion Show Location and Crowds Please Exhibitors

To an extent not equalled in recent years, exhibitors at the 1960 Corrosion Show at Dallas were vehemently enthusiastic about almost everything that happened at the show. This attitude was the result, in large measure, of the efficient work of the decorator, the excellent facilities available at the Dallas Municipal Auditorium, and the convenient location of the show with respect to the meeting rooms.

No small measure of credit is due to the exhibitors themselves who provided what many believe to be the most consistently interesting exhibition NACE has ever held. In the well-lighted auditorium, the large aisles, good ventilation and cooperation of auditorium personnel helped maintain the general atmosphere of satisfaction.

Attention to the show was distributed over the whole three days with the natural climax during the Tuesday afternoon open period when large crowds attended.

Unseasonably cool weather worked to the disadvantage of outdoor exhibits. There was no rain during the three days of the show, however.

The 1960 Show was the first sponsored by NACE to be sold out six months in advance. R. J. Huff, Jr., NACE Exhibits Manager, pointed out that the 1961 show in Buffalo will be smaller than the Dallas show. Information about the 1961 event will be mailed in May.

## Over 10,900 Attend '59 NACE Meetings

More than 10,900 persons attended 262 meetings held by or with cooperation of NACE during 1959, according to a report by H. C. Van Nouhuys, Marietta, Ga., chairman of the Regional Management Committee. Mr. Van Nouhuys said that these meetings included 11 short courses, a record number during one year.

Section publicity showed a 17 percent increase in 1959 over 1958, he said while the Canadian Region led all other NACE Regions in increased membership with a gain of 12.4 percent over the preceding year. North Central region was second in net gain with 5.5 percent and Western Region third with 3 percent. Northeast, Southeast and South Central Regions showed either losses or nominal increases.

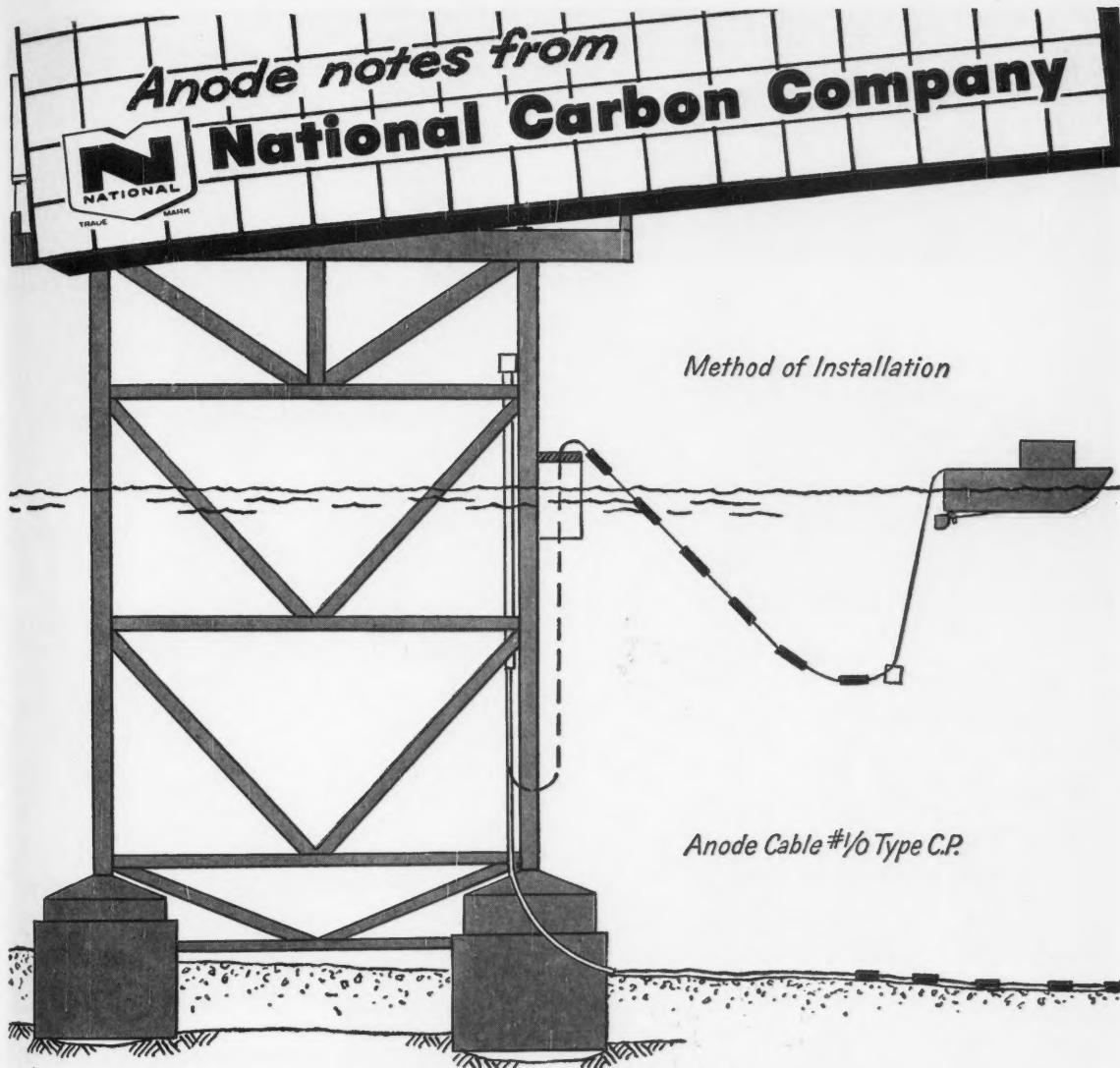
Net association's membership increase for the year was 2.8 percent.

## Former Executive Secretary Adds Corporate Members

A. B. Campbell, retired executive secretary of NACE, told the board of directors about some of the work he is doing in trying to add corporate members. Mr. Campbell has been applying himself to this activity during the past 18 months and reports that he has made some progress and expects to make more.

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## PROTECTION OF A \$4,750,000 OFFSHORE DRILLING RIG

Corrosion engineers of a large oil company decided to use impressed current cathodic protection on the underwater foundation of an offshore drilling rig.

Six strings of 10 "National" NA Graphite Type QA Anodes\* were used to provide a distributed anode bed. They were assembled on 1/0 C.P. cable at fifteen foot spacings. Anode strings were placed radially around the plat-

form on the sea bottom with the first anode approximately 200 feet from the platform base. The anode strings are held in place by 12"x12"x16" concrete anchor blocks at the end of each string.

"National" NA Graphite Anodes were selected because of proved long anode life (about 0.1 lbs./amp. year consumption in free moving salt water) and low initial cost.

\*Anodes were sold by The Vanode Company, Pasadena, California



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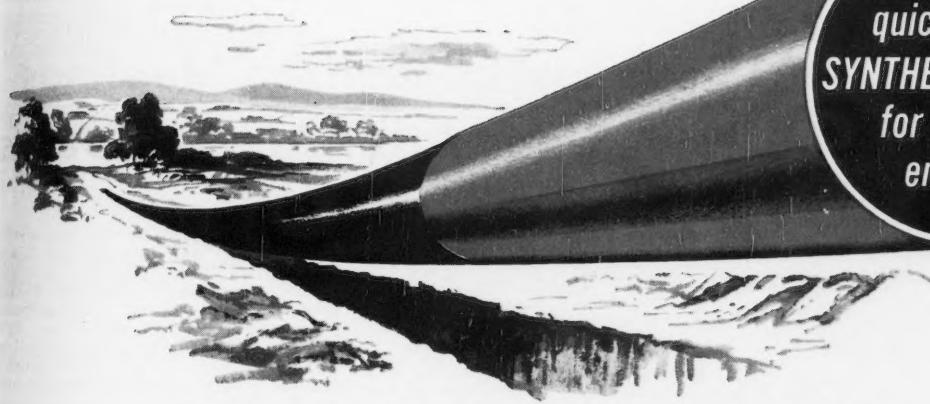
### CONFERENCE SNAPSHOTS

(1) "We didn't make any inhibitor tests . . ." was part of this group's conversation outside a meeting of T-1. They are Gordon L. Nunn, Tidewater Oil, Houston, Texas, A. J. Orr, Jr., Corrosion Engineering Service Co., Houston, and John D. Kroner, Plastic Applicators, Inc., Shreveport, La. (2) The conference progressed so smoothly that General Chairman J. Gordon Meek of Dallas (seated) and E. J. Simmons could take time to read the comics. (3) J. A. Schauers of Atlantic Pipe Line, Philadelphia, Pa., is shown deciding which technical meeting he will attend. Looking on are T. Williamson, James E. Prater, Jr., Atlantic Pipe Line, Dallas, Texas, and George H. Silkworth of El Paso Natural Gas Co., Salt Lake City, Utah. (4) More discussion is being held by George P. Simon, William F. Brindley, Roger G. Christman (all of Westinghouse, Pittsburgh, Pa.) and Kurt Jakobson of the Martin Co., Baltimore, Md. (5) Thanks to the Dallas Chamber of Commerce helpers, registration lines were not long. (6) Edward G. Brink, an NACE Director, is shown with incoming NACE President George Best at the Corrosion Show. (7) Another group at the Corrosion Show. (8) F. Parker Helms of Union Carbide Chemical Co., Texas City, Texas, presents his paper at the Protective Coatings Symposium. Symposium Chairman R. E. Gackenbach of American Cyanimid Co., New York, N. Y. is also shown. (9) Another informal discussion group: Ernest O. Kortner, Signal Oil & Gas Co., Los Angeles, Cal., Raymond A. Oliver, Pure Oil Co., Lafayette, La., William H. Edwards, Superior Oil Co., Houston, Texas, Edward Schaschl, Pure Oil Co., Crystal Lake, Ill., and Del Perkins, Termec, Maracaibo, Venezuela. (10) Part of the members of T-21 are shown: Dick Hoehl, Trenton Corp., Ann Arbor, Mich., Ted Kennedy, Jr., Trenton Corp., Ann Arbor, Mich., J. W. Moffett, Ohio Oil Co., Martinsville, Ill., and Harold Bussell, Dow Chemical Co., Lake Jackson, Texas. (11) Mrs. J. C. Spalding of Dallas was one of the 150 women registered for the conference. (12) The NACE booth at the Corrosion Show had many visitors looking over publications available. (13) Mrs. Ted Skaggs of Birmingham, Ala., was seen at the Fellowship hour.



# REILLY REDHEAD

**NEW  
quick-drying  
SYNTHETIC PRIMER  
for coal-tar  
enamels**



**Translucent red color permits visible detection of any areas  
missed in cleaning...any areas not covered by primer...any areas  
not covered by enamel!**

New Reilly Redhead, a quick-drying synthetic primer for hot coal-tar enamels, is compounded specifically for difficult drying conditions where initial high bond strength is mandatory. Reilly Redhead provides initial bond strengths up to several times greater than typical non-synthetic primers and fractionally greater strength than

Reilly coal-tar base primers X-1 and X-10.

Reilly Redhead dries to touch in 5 to 10 minutes after application to metal surfaces at 70°F. where relative humidity does not exceed 70% and is free from organic solvent vapors. It dries hard, ready to accept enamel, within two hours under the same conditions.

*Other important advantages are:*

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- Conforms to specifications under consideration by A.W.W.A. and governmental agencies.
- Performs advantageously with all Reilly plasticized hot application enamels.
- Is available in 55, 5 and 1-gal. containers, in carloads, carloads mixed with enamel, or less than carload, shipped from Indianapolis, Ind. or Lone Star, Tex.

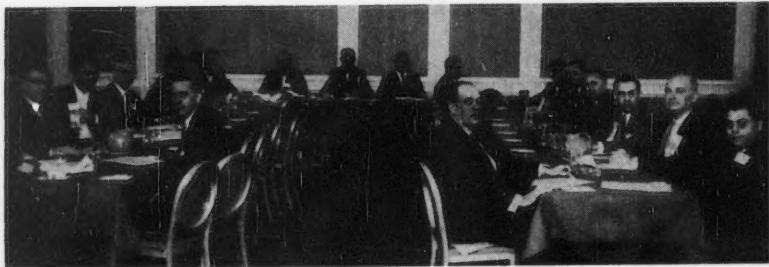
Write today for complete information on new Reilly Redhead, the quick-drying high bond strength synthetic primer. See how you can use Reilly Redhead profitably in your operations.

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TAR PRODUCTS DIVISION 1615 Merchants Bank Building

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NEW NACE BOARD meets for the first time on the morning of Friday, March 18 at the NACE Dallas Conference. President George E. Best, Manufacturing Chemists' Association, Washington, D. C. is presiding.

## Technical Advisers On NACE Committees Doubled in 5 Years

In a discussion of the activities of NACE technical committees at a meeting of the association's board of directors Sunday March 13 at the 16th annual conference in Dallas, T. J. Maitland, American Telephone and Telegraph Corp., New York, chairman of the Technical Practices Committee observed that technical advisers associated with NACE committees have more than doubled in number in five years. This increase has been in spite of a change in procedures five years ago when NACE membership was made a prerequisite to membership in committees.

Mr. Maitland also reported that it appears that many meetings of technical committees apparently are discussion sessions only and that little or no

actual technical results are being realized from them.

During the board discussion of technical committees it was suggested that committee reports should be revised from time to time in order to take into account recent developments in materials and techniques.



CREDIT TO WORKING MEMBERS of the conference and corrosion show committee was given by J. Gordon Meek, Metal Goods Corp., Dallas, general conference chairman for the 16th conference at the annual banquet.

## Technical Committee Meetings Have Good Attendance at Dallas

Attendance was good at most of the 65 technical committee meetings held at Dallas during the 16th Annual NACE Conference March 14-18. Actions and discussions of some of the committee meetings are given below.

### T-1

Group Committee T-1 (Corrosion in Oil and Gas Well Equipment) had standing room only at its meeting Monday, March 14, in the Adolphus Hotel. Reorganization of T-1, begun at the South Central Region Conference last October in Denver, was explained fully to the members.

### T-6F

Five task group reports were given at the meeting of Technical Committee T-6F (Protective Interior Linings, Application and Methods). T-6F Chairman W. P. Cathcart, Tank Lining Corp., Pittsburgh, Pa., heard reports from the task groups on curing, surface preparation, inspection, safety and application.

### T-4A

A total attendance of 75 was counted at the meeting of T-4A (Effects of Electrical Grounding on Corrosion). C. L. Mercer was nominated vice chairman. I. C. Dietze, chairman of T-4A-3 (Methods and Materials for Grounding) gave a report from this task group. A committee was set up to form a new task group on grounding practices. A. F. Minor was appointed chairman of this new group to be designated as T-4A-4.

### T-3H

Informal discussion with good participation by the 40 attending members was experienced at the T-3H (Tanker Corrosion) meeting with Chairman W. S. Quimby presiding. Several phases of tanker corrosion including controversial issues were included in the discussions.

### T-9B

Proposed changes in creosote specifications were discussed at the Dallas meeting of T-9B (Preservatives and Their Appraisal). Consensus of the group's opinion was that the proposed specification changes are in the right direction. Use of the assay method to check creosote retentions is gaining popularity with consumers of creosoted piling. Many creosote treaters in the southern states are not equipped to provide the retentions required for marine piling.

(Continued on Page 58)

## Lapel Pins



Approx.  
Size

Approximately 7/16 inches high, gold, inlaid with bright red enamel background to "NACE" and deep blue enamel background to words "CORROSION CONTROL." Ruby center.

For Association Members Only

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### MARINE COATINGS

T-1M Suggested Coating Specifications for Hot Application of Coal Tar Enamel for Marine Environment. A Report of NACE Technical Unit Committee T-1M on Corrosion of Oil & Gas Well Producing Equipment in Offshore Installations. Publication No. 57-8. Per Copy \$.50.

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Remittance must accompany all orders for literature the aggregate cost of which is less than \$5. Orders of value greater than \$5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 65¢ per package to the prices given above for Book Post. Register to all addresses outside the United States, Canada and Mexico.

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or encasing in concrete

Only KRALOY, yes *only* KRALOY, among all plastic conduits, now carries U-L listing — another first for KRALOY! High impact KRALOY PVC (Polyvinyl Chloride) RIGID CONDUIT... the *lifetime* conduit... the *perfect* conduit... won't rust, won't pit, won't corrode, won't support combustion, is non-magnetic and non-sparking. KRALOY needs no paint, no coating, needs no lining, and its mirror-smooth inside wall makes fishing easier than with any other type of conduit. Add these outstanding features to KRALOY's extreme light weight (see chart below) and you have the ideal conduit... ideal for direct burial and slab work. Installation costs can be cut *drastically* with light weight KRALOY PVC RIGID CONDUIT. Consider the dollars to be saved in handling and installing U-L listed KRALOY CONDUIT Cat. No. KE-1058 versus steel, *versus even aluminum conduit*:

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sold only through wholesale  
electrical supply houses.

#### NOTE WEIGHT COMPARISON — KRALOY PVC vs. ALUMINUM vs. STEEL CONDUIT

U.L. required minimum weight per 100 ft. including couplings, lbs.

Trade Size	1/2"	3/4"	1"	1 1/4"	1 1/2"	2"	2 1/2"	3"	3 1/2"	4"	5"	6"
<b>KRALOY PVC</b>	15.0	20.0	29.0	40.0	47.0	63.0	101.0	131.0	159.0	187.0	253.0	326.0
<b>ALUMINUM</b>	27.4	36.4	53.0	69.6	86.2	115.7	182.5	238.9	287.7	340.0	465.4	612.9
<b>STEEL</b>	79.0	105.0	153.0	201.0	249.0	334.0	527.0	690.0	831.0	982.0	1334.0	1771.0

For complete information on KRALOY PVC CONDUIT and installation directions, mail the coupon or write to Kraloy Plastic Pipe Co., Inc., 402 W. Central Ave., Dept. C-4, Santa Ana, Calif.



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402 W. Central Ave., Santa Ana, Calif.

Gentlemen: Please send me your new Brochure on KRALOY PVC CONDUIT which gives complete information and installation directions.

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NO CONDUIT REPLACES

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## Review Committee Seeks To Publish High Quality Papers

Higher standards for technical papers published by NACE is the aim of the Editorial Review Subcommittee of the association's publication committee. At a meeting in Dallas during the 16th conference, the committee decided to be more discriminating in selecting papers sent out for review to the committee. Members reported that some of the papers sent out were so poor that review was not merited.

Operations of the committee were discussed and some changes in administrative procedure were recommended.

Present for the meeting were F. M. Reinhart, National Bureau of Standards, Washington, D. C., chairman; Ivy M. Parker, Plantation Pipe Line Co., Atlanta, editor of CORROSION; Harry J. Keeling, consultant, Los Angeles; Mark F. Adams, Washington State College, Pullman; R. S. Treseder, Shell Development Co., Emeryville, Cal., publication committee chairman and John J. Halbig, Armco Steel Corp., Middletown, Ohio.

Western Region's 1960 Conference will be Oct. 6-7 at the Sheraton Palace Hotel in San Francisco, Cal.

THIS FULL SIZE TANK shown by Black, Sivalls and Bryson permitted engineers to get an estimate of its usefulness by actually examining it inside and out. (Middle) This machine demonstrated application of Polyken tape on small diameter pipe and (bottom) Owens Corning Fiberglas demonstrates application of a water resistant material designed for use on ceramics.

## Practical Applications Described For Thin Film Corrosion Indicators

Practical applications of newly perfected thin film corrosion indicators were described by David Roller, Magna Products Inc., Santa Fe Spring, Cal., in his paper "Development of Thin Metal Film Corrosion Indicators." Gibson H. Rohrbach of the same company is co-author of the paper presented during the 16th conference at Dallas.

The indicators, which consist of microscopically thin films of metals vacuum-deposited on appropriate substrates, can be used as extremely sensitive indicators of impending corrosion by atmospheric pollutants, moisture, and other corrosives. Two types of indicators are available. One gives a visual warning of corrosion damage by changing color, while the others, supplied in several configurations, give like warnings through electrical resistance measurements.

The detectors, which were developed under research grants made by the United States Air Force, are being marketed under the trade name "Magna-

film." The principal advantage these indicators offer, Mr. Roller pointed out, is that they give a warning of impending corrosion before it happens and thus permit preventive action to be taken.

Field tests are being made in several thousand units of packaged equipment. Small warning meters make possible assessment of changes in the remote interiors of large packages while they remain hermetically sealed. This is accomplished by a long-lived self-powered warning meter in which a light goes on as soon as corrosion has begun to attack the indicators connected to it.

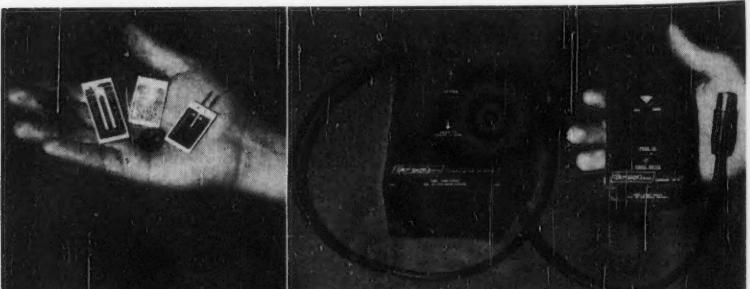
Mr. Roller also reported that by using a calculator the extent of corrosion measured by the increasing resistance of the film could be converted into micro-inches a year. The order of penetrations thus detectable were in milionths.

Versatility of the new indicators is sure to bring about their widespread use in industry, he said.



### FOCUS on COATINGS

Five outstanding articles on coatings will be published in the Technical Topics Section of the June issue of CORROSION. A wide variety of coatings topics will be included.



CORROSION MEASUREMENTS in millionths of an inch can be made with these Magnafilm indicators. From left to right they are, visual type, tempera-

ture compensated electrical resistivity type, and three configurations of the non-temperature com-

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# Oil Industry Has Large Stake In Nation's Corrosion Control

The oil industry's stake in corrosion control was emphasized at the Dallas conference during a luncheon of NACE corporate member representatives. William C. Kinsolving, president of Sun Pipeline Company and Sun-Canadian Pipeline Company, Philadelphia, told over 150 present that no industry in the United States has a greater stake in corrosion control than does the oil business. The significance of NACE's work to the oil business becomes evident when the industry's 200,000 miles of petroleum



## TECHNICAL REPORTS on

### HIGH PURITY WATER CORROSION

Symposium on Corrosion by High Purity Water by Committee T-3F on High Purity Water which includes:

Introduction to Symposium on Corrosion by High-Purity Water by John F. Eckel.

Corrosion of Structural Materials in High-Purity Water by A. H. Roeback, C. R. Breder and S. Greenberg.

Corrosion Engineering Problems in High-Purity Water by D. J. DePaul.

The Importance of High-Purity Water Data to Industrial Applications by W. Z. Friend. Per Copy ..... \$1.50

Symposium on Corrosion by High Purity Water. Five Contributions to the Work of NACE Technical Committee T-3F on High Purity Water. Pub. 57-22.

Measurement of Corrosion Products in High Temperature, High Pressure Water Systems by A. S. Sugalski and S. I. Williams.

Corrosion of Aluminum-Nickel Type Alloys in High Temperature Aqueous Service by F. H. Krenz.

Corrosion of Aluminum in High Purity Water by R. J. Lobisinger and J. M. Atwood.

The Storage of High Purity Water by Richard R. Dlesk.

Water Conditions for High Pressure Boilers by D. E. Voyles and E. C. Fiss. Per Copy ..... \$1.50

Symposium on Corrosion by High Purity Water. Four Contributions to the Work of NACE Tech. Comm. T-3F on High Purity Water. Pub. 58-13. Per Copy ..... \$1.50

Corrosion Behavior of Zirconium-Uranium Alloys in High Temperature Water, by W. E. Berry and R. S. Peoples.

Corrosion and Water Purity Control for the Army Package Power Reactor by R. J. Clark and A. Louis Medin.

Removal of Corrosion Products from High Temperature, High Purity Water Systems with an Axial Bed Filter by R. E. Larson and S. L. Williams.

Some Relations Between Deposition and Corrosion Contamination in Low Make-up Systems for Steam Power Plants by E. S. Johnson and H. Kehmara.

Remittances must accompany all orders for literature the aggregate cost of which is less than \$5. Orders of value greater than \$5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 60¢ per package to the amount given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

## NATIONAL ASSOCIATION OF CORROSION ENGINEERS

1061 M & M Bldg. Houston 2, Texas

## NACE Adopts Green As Official Color

Persons of Irish ancestry in NACE will be especially glad to learn that green is now the official color of the National Association of Corrosion Engineers. The NACE board decided to adopt green as the official color of the association during its initial meeting at Dallas during the 16th conference.

The decision was made in time for St. Patrick's Day and partially in recognition of the connection between green and NACE that has been built up over the years by the consistent use of green on CORROSION's cover and as a color of covers of association publications.

## Technical Committees—

(Continued From Page 54)

### T-6R

J. H. Cogshall, chairman of T-6R (Protective Coatings Research) reported that the annual report of corrosion testing activities currently being conducted by other societies and association groups may be useful to members of T-6.

### T-2E

Primary topic of discussion at the T-2E (Internal Corrosion of Product Pipe Lines and Tanks) meeting was on data received on the T-2E and T-8 questionnaires on corrosion losses in refined products storage tanks. Data have been received for 59 tanks. Eighteen percent showed losses of 0.10 inches or more. Of these, 73 percent were aviation gasoline tanks. Losses in all these tanks but one were greatest in the upper half of the tanks.

Thirty-two percent showed losses of 0.05 to 0.09 inches. Forty-two percent of these were aviation gasoline tanks. Again greatest losses were in the upper half of the tanks.

Additional work must be done before a standardized procedure can be proposed for the modified ASTM rust test.

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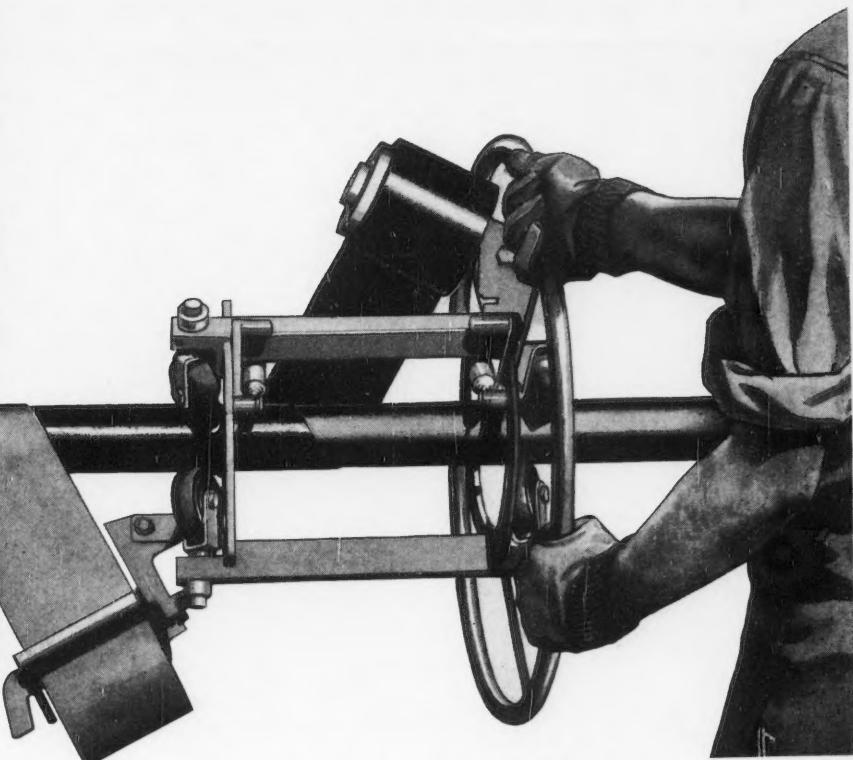


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The 1960 South Central Region Conference will be held October 25-28 at the Mayo Hotel, Tulsa, Okla.

# AN EASY WRAP-ON WAY TO STOP PIPE CORROSION



## Chasekote\*

Pressure-Sensitive Polyethylene Tape has an extra-heavy adhesive mass that clings to clean pipe on contact . . . makes a perfect, permanent bond that rustproofs, waterproofs and insulates pipe against all types of corrosion.

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Protective Overwrap is a tough, wrinkle-free outerwrap that shields against abrasion, wear, and physical damage to the tape. Wraps simultaneously with the tape. Superior to rag and felt warps, yet far lower in cost.

\*Trade name of Chase & Sons, Inc., long-famous for protective and insulating tapes for electrical wire and cable



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**Carboline's new zinc filled  
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- Water-insoluble 20 minutes after application.
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Carbo Zinc 11 can be used with topcoat or as primer with vinyl, Hypalon or inorganic renewable topcoat for color. Apply with brush or spray. In non-immersion service, it can be applied over a commercial blast surface. It's economical, too . . . low material, application and maintenance costs.

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## Superior Facilities Of Dallas Auditorium Helped Attendance

Attendance was good at almost all of the technical committee meetings held at Dallas during the 16th Annual Conference. The superior facilities at the Dallas Memorial Auditorium contributed to the satisfactory meetings in large measure. Rooms were, with a few exceptions, sufficiently large for the sessions and were adequately lighted and ventilated.

The Dallas arrangements committee provided the required extra equipment used in some presentations at the meetings.

As could be anticipated from the geography, meetings of committees associated with the petroleum industry were particularly well attended. This was true also of those in the coatings and plastics fields.

The reorganization proposed by T-1 on Corrosion of Oil and Gas Well Equipment was approved by the board of directors.

### Color Slide-Talk on 1960 Corrosion Show

A 35-mm color slide show of about 100 slides of exhibits at the 1960 Corrosion Show at Dallas has been prepared and is available for showing at NACE Section meetings. The show, which has a commentary on specific items exhibited, is available on a first-come, first-served basis on application to G. A. Rolak, NACE Central Office.

The show provides an opportunity for NACE members who were unable to visit the Corrosion Show personally to see some of the equipment on display. While the presentation does not include all the exhibits, it will give a good idea of the show. Presentation time should vary between an hour and an hour and a half.

### Water Conversion Costs

Future costs for sea water conversion may be lowered to less than one dollar per 1000 gallons for large installations. In 1950, conversion cost estimates were as high as five dollars per 1000 gallons. Improved processes and equipment reduced that cost to about two dollars. Additional improvements in distillation processes and development of a freezing process may justify the lower estimates for the future.

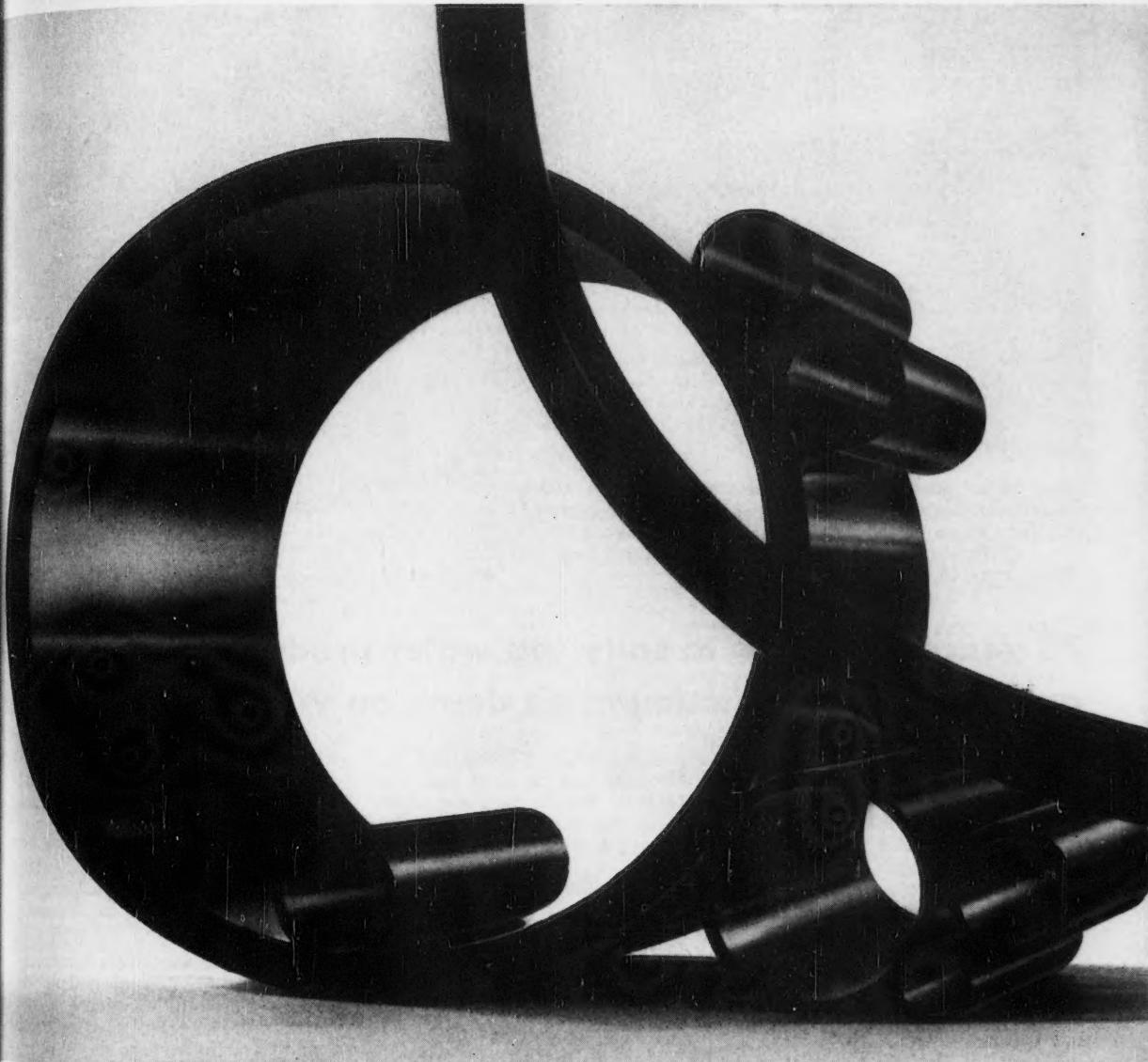
### Nickel Consumption

Despite the steel strike, U. S. nickel consumption in 1959 increased about 35 percent over 1958, according to John F. Thompson, chairman of the International Nickel Company of Canada's board of directors. Nickel consumption in 1959 for the free world exceeded 400 million pounds—an increase of about 25 percent over the 1958 total consumption.

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It's a fact! Now you can buy plastic pipe that's virtually trouble-free! It's made of A-C Pipe Compound, a super-strong polyethylene resin designed to last 50 years—and actually covered by manufacturers' *warranties* for 20 years or more!

This pipe offers tremendous surge strength and complete freedom from slitting or pinholing. It is the *only* flexible plastic pipe available today that is entirely free from environmental stress cracking.

Note how this glossy premium pipe is shiny

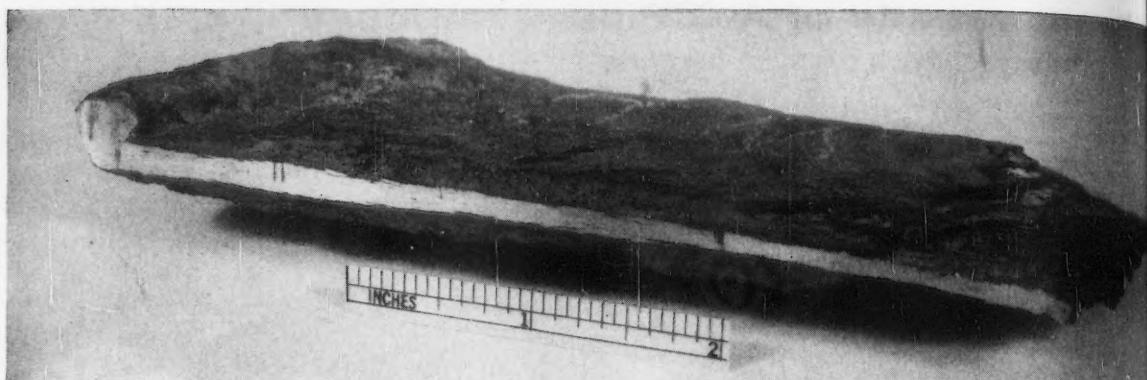
*inside* as well as *out*! It will not rust, rot, or corrode, and has excellent resistance to acids and alkalies. It withstands sub-zero temperatures, is lightweight, flexible, easy to install.

Pipe made of A-C Pipe Compound assures lasting performance in jet wells, water transmission, chemical and food processing. N.S.F.-approved and nontoxic, it's suitable for dozens of uses in home, farm or industry. For the names of manufacturers, or for sample test lengths, write us today.



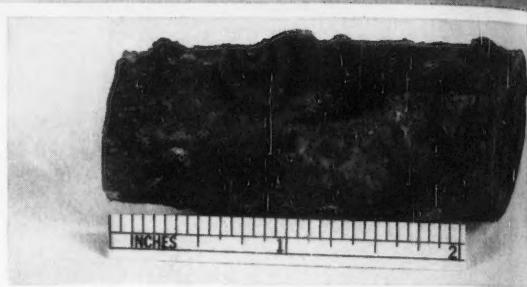
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Dept. 570-BQ, 40 Rector Street, New York 6, N. Y.



**Wrought Iron plate cut by hand from fire curtain of Marina Yacht Harbor Pier, San Francisco, California**—The dense and compact nature of Wrought Iron's protective scale is clearly evident. Its adherence is permanent: on plate after 26 years, and on tubular goods after 73 years of salt water service.

**Steel tie rod after cleaning and measuring adjacent to deck where paint was still intact**—Note chipped and spalled appearance of scale on rimmed steel tie rod. This tie rod was originally 1" material. When cleaned of rust, the rod measured 0.481", 0.490" and 0.697" on three different diameters.



## 73 years' exposure to salty sea water produced only 3 mils/year maximum pit depth on Wrought Iron

A structural inspection of the Marina Yacht Harbor Piers in San Francisco, Calif., revealed once again, the decided superiority of Wrought Iron over steel in salt water environment.

Maximum pit depth on Wrought Iron after 73 years' exposure to sea water and sea air was 210 mils: approximately 3 mils/year.

Steel similarly located, showed perforation of  $\frac{1}{4}$ " plate after 18 years: approximately 14 mils/year. Severance of one-inch steel rods occurred in 26 years.

No case of Wrought Iron perforation was evident. Either in  $\frac{1}{4}$ " plate over a period of 26 years, or in tubular goods over a period of 73 years.

Corrosion researchers reported the following detailed findings in their structural inspection:

- Scale on Wrought Iron was heavy and dense. Scale adhered so tightly that a diamond pattern hammer was required to remove it. Scale on steel was equally thick. But it was so friable that light hammering was sufficient to remove it.

- Some scabbing occurred on Wrought Iron at mean high tide (MHT). But, in every case where a scab was hammered off, a dry inactive pit was found.
- At MHT, the maximum pit depth of Wrought Iron was 210 mils. Steel at the same position showed depths of as much as 360 mils. This occurred even though the steel had been in service less than 26 years. Wrought Iron had already withstood 73 years' service.
- In the area of MHT, where the maximum Wrought Iron pit depth was 210 mils in 73 years, the  $\frac{1}{4}$ " (250 mils) steel plate had perforated in less than 18 years.
- Wrought Iron,  $\frac{1}{4}$ " thick, adjacent to the  $\frac{1}{4}$ " steel plate which had perforated in 18 years, showed a maximum loss of 187 mils in 26 years. This occurred in spite of the fact that the Wrought Iron had been subjected to extreme crevice corrosion not obtaining on the steel.

The most casual observation reveals use of two different materials in these docks. The Wrought Iron, after 73 years' exposure to salt water and salt air, is today in much better condition than the steel installed 26 years ago.

**A complete inspection report of the Marina Yacht Harbor Piers is available on request. Write A. M. Byers Company, Clark Building, Pittsburgh 22, Pa.**



**BYERS 4-D WROUGHT IRON**

TUBULAR AND FLAT ROLLED PRODUCTS

Corrosion costs you more than Wrought Iron

MINIATURE  
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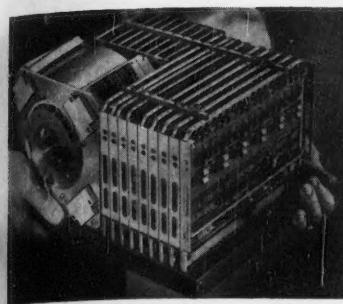
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**MINIATURE COMPUTER** weighing only 32 pounds and occupying 0.55 cubic feet has been developed for use in the Centaur guided missile. Designed with transistors, the small digital computer will perform necessary guidance computations and provide steering signals to the missile's control system.

## Midget Computer Built for Moon Trip

A digital computer weighing only 32 pounds and occupying only 0.55 cubic feet is being built for use in the Centaur missile, designed to place a 730-pound payload on the Moon.

Silicon transistorized throughout, the computer will be integrated with the Centaur's guidance system to accept inputs from the inertial platform. It will perform the necessary guidance computations and provide steering signals to the Centaur's control system.

The Centaur also will be used to send instrumented space research and communications satellites into 300 to 22,000 mile orbits. It consists of a modified Atlas first stage rocket coupled to a 15,000 pound thrust liquid oxygen and hydrogen second stage under development by Convair and Pratt & Whitney. The third stage liquid engine will be developed by Jet Propulsion Laboratory. The Centaur program is sponsored by the National Aeronautical Space Administration.

The computer was developed under a subcontract to Librascope, Inc., Glendale, Cal., by Minneapolis Honeywell, developers of the Centaur's inertial guidance system.

## Plastic Replaces Brass For Water Faucet Parts

Delrin acetal resin is being used to replace brass parts of the cartridge mechanism in a new self-seating faucet. These parts are injection molded as finished units, thus saving the cost of machining that was necessary with brass.

Delrin, a high strength polymer which has metal-like properties, has corrosion resistance characteristics and will remain free from hard water deposits, according to the faucet manufacturer, Kel-Win Manufacturing Co., Inc., Richmond, Va. Delrin is a new thermoplastic material made by Du Pont.

Long service life is expected because of the strength, dimensional stability, stiffness and resistance to heat distortion of Delrin. Du Pont tests indicated that shower heads of this new material should have a useful life of 20 years.

NACE's 17th Annual Conference and 1961 Corrosion Show will be March 13-17, 1961, at the Hotel Statler in Buffalo, New York.



## Defense Department Establishes Center For Plastics Data

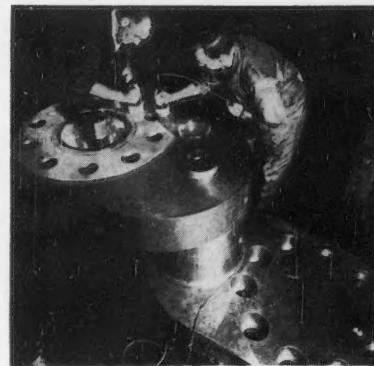
The Department of Defense has established a Plastics Technical Evaluation Center at Picatinny Arsenal, Dover, N. J. The center is designed to collect and evaluate data on plastics, provide technical assistance on plastics to military contractors and suppliers, prepare and issue reports and stimulate advances.

A group of specialists will be employed at the center to function in four areas: structural uses; electrical and electronic uses, packaging and mechanical uses. The specialists will collect and disseminate data in the field and will concentrate on evaluation of the information acquired.

Information collected by the center will be disseminated in answers to specific questions and publications. Services of the center are available without cost to anyone who has a legitimate interest in furthering the intelligent use of plastics in military applications.

The premise on which the center is established is that, while the research scientist has the time and resources to make literature searches for new information, the engineer has neither. As a consequence, the engineer tends to continue using materials and methods with which he is familiar. Furthermore, according to John R. Townsend, special assistant to the director of Defense Research and Engineering, speaking to the Society of Plastics Engineers in December, 1959, "To the scientist, the production of information is an end in itself. He downgrades his own usefulness if he tries to anticipate the usefulness of his findings. The scientist is fond of the prestige of being the first to publish a new discovery. Engineers are much less

(Continued on Page 66)



**FRETTING CORROSION** on highly stressed engine parts of ocean-going vessels such as the diesel crankshaft above is prevented by an electroplated tin coating which will also serve as a gasket. The

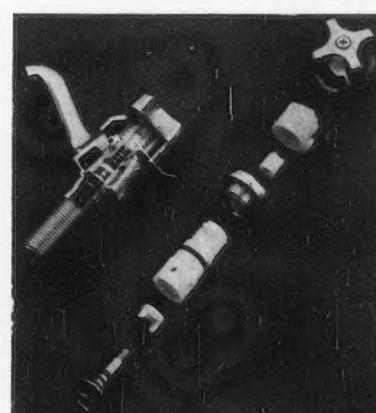
## Tin Plate Prevents Fretting Corrosion

Special preventive maintenance plating techniques are providing extended service life for highly stressed engine parts on ocean-going vessels. The plating process can be conducted in the ship's engine room.

Tin is electroplated onto engine parts such as crankshafts to serve as a gasket and to prevent fretting corrosion.

The Dalic portable selective plating process developed in France is used. The anode pad is saturated with a rapid plating solution and rubbed over the areas to be plated (cathode). This process is being handled in the United States by Sifco Metachemical, Inc., 935 E. 63rd St., Cleveland, Ohio.

Preventive maintenance plating of the diesel crankshaft shown in the illustration required only 9 man hours.



**THERMOPLASTIC PARTS** are being used to replace brass in a self-seating faucet. Made of Du Pont's Delrin, the parts have metal-like properties plus the corrosion resistance of plastics.

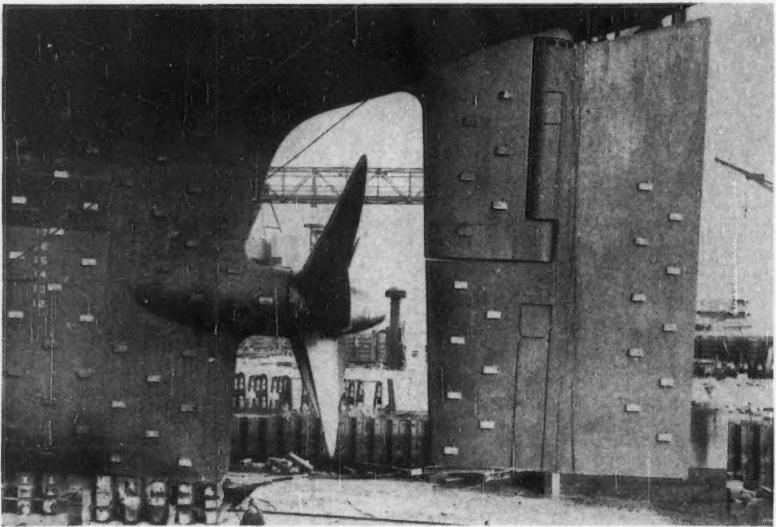
## Giant Filter Removes Atomic Fallout in Water

A special giant filter tank has been installed to remove radioactive contaminants in a paper manufacturer's process water. Radioactive fallout from atomic and thermonuclear bomb tests in the USA and Russia can contaminate river water to such extent that the water is unusable for manufacturing certain papers used by manufacturers of light sensitive photographic materials.

Even the most minute amounts of radioactivity in special papers used in packaging photographic materials can cause defects in the photographic films.

The filter was built at the Knowlton Brothers plant at Watertown, N. Y., on the Black River.

There were 311,758 stockholders in one U. S. steel company in 1958. These stockholders included men and women from all walks of life in every state in the nation.



ZINC ANODES patterned around the stern of the new 70,000-ton supertanker "Princess Sophie" were designed to control stern area corrosion. Without these hull anodes, the vessel's plates would have corroded because of the electrochemical reaction between the steel hull and the 37-ton bronze propeller. The tanker's interior cargo-ballast tanks also were fitted with a specially designed magnesium anode cathodic protection system. The "Princess Sophie" is considered the largest bulk carrier ever built in the United States; she has a capacity of 27,200,000 gallons of crude oil or petroleum products and cruises at an average speed of 16½ knots.

## Waste Treatment Meeting Scheduled for April 20-22

Forty-five technical papers including 10 from England, Germany, Holland, Japan and Sweden will be given at the Third Conference on Biological Waste Treatment at Manhattan College April 20-22.

Devoted to new advances in biological oxidation and oxygen transfer in the disposal of waste from industry and municipalities, the conference is made possible by a grant from the Department of Health, Education and Welfare. Additional information can be obtained

from W. Wesley Eckenfelder, Civil Engineering Department, Manhattan College, New York 71, N.Y.

## Small Aluminum Pipe Laid At 3 mph by New Method

A fast and simple method for laying small diameter aluminum pipe was used in a recent test at Denver, Colo. Automatic pipe-laying equipment dug a 42-inch deep trench, unreeled and laid 2-inch Schedule 5 aluminum pipe and back-filled—all in one uninterrupted operation. Installation rate was approximately three miles per hour—faster than the usual rate for small piping.

## NEW Casing Seal! WMSON "U" SEAL

MAINTAINS TIGHT SEAL EVEN WHEN DRAG SECTION CHANGES POSITION IN CASING

This versatile and flexible "U"-Seal offers NEW ECONOMY in casing seals.

The special demonstrator pictured shows the flexibility of the "U"-Seal. The pipe position changes, but the seal remains tight. A special fungus and bacteria retardant is added to the compound for extra protection. Seals available in sizes 2" and larger.

### VERSATILITY OF INSTALLATION

The fully reversible "U"-Seal is easy to install in the new "U" shape or in Wmson's familiar "Z" shape. A screwdriver is the only tool needed to tighten the two stainless steel bands.

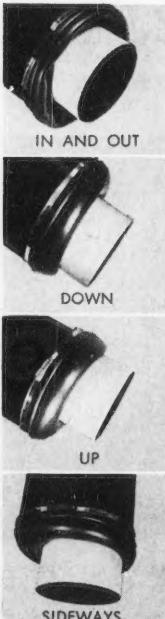
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YOU CAN SEAL WITH A "U"-SEAL



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REPRESENTATIVES AROUND THE WORLD



## First International Corrosion Congress To Meet in London

The First International Congress on Metallic Corrosion is scheduled to be held April 10-15, 1961 in the Imperial College of Science and Technology, London. L. Kenworthy, chairman of the Corrosion Group of the Society of Chemical Industry is chairman of the technical program.

The program is being organized under the following schedule: 1. Corrosion principles and fundamentals. 2. Papers

### MEMBERSHIPS AVAILABLE

Persons interested may apply for membership in the congress by filling out an application card and returning it with 5 pounds sterling to Francis J. Griffin, Secretary; 1st Int. Cong. Metallic Corrosion, 14 Belgrave Sq., London, S.W.1. Cards may be obtained from Mr. Griffin or an application including the full name, mailing address together with the fee may be sent to him.

relating to particular metals. 3. Papers relating to environments: Atmospheric, water, soil, other media, high temperature. 4. Papers relating to methods of protection: Coatings metallic and non-metallic, inhibitors, cathodic protection and others. 5. General papers dealing with a range of materials or conditions. 6. Economics and design. 7. Methods of testing and 8. Education.

## Metal Solidification to Be August Symposium Topic

A symposium on engineering aspects of solidification of metals will be held August 22-26 at Massachusetts Institute of Technology. Theoretical lectures will lead to practical examples of how the properties and reliability of castings and forgings can be enhanced by improved control over the solidification pattern of the casting or ingot. Additional information can be obtained by writing Office of the Summer Session, MIT, 77 Massachusetts Ave., Cambridge 39, Mass.

## Cost Engineers to Meet

The 1960 national meeting of the American Association of Cost Engineers (AACE) will be held June 13-15 at the Rice Hotel, Houston, Texas. The technical program will include papers and work shop sessions on cost estimating and economic evaluation.

## Defense Department —

(Continued From Page 65)

moved by this incentive. Characteristically, engineering developments result in an accumulation of notebooks, records, working papers, internal staff reports, and unrecorded data in the head of the engineer. These data do not find their way into the published literature for a year or two, if ever.

Over 20 million dollars was paid in 1959 to 39,000 employees of one company through the company's savings plan.

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## Research May Increase Lanthanon Compound Uses

Lanthanon compounds (rare earth oxides) may have increased uses in the ceramic industries because of recent research and developments which help to reduce cost and increase the supply. Melting points of these oxides are high enough for possible refractory uses such as crucibles for melting metals, glass or enamel compositions. Nuclear ceramics and electronic ceramic applications also are being found for the lanthanons.

## Correction Given for Table On International Prefixes

A corrected form of the table of new international prefixes to be used for denoting multiples is given below. The table was published on Page 39 of the February issue of CORROSION with an error in the line next to the bottom. The superscript number in that line should have been -9 instead of -8. The corrected table of prefixes, symbols and their pronunciation are given below.

In addition to the eight numerical prefixes in common use, which are given in the table below, the committee expanded the list by adding the four prefixes marked with an asterisk. Thus, for example,  $10^{-12}$  farad is called 1 picofarad and is abbreviated 1 pf.

Multiples and Sub-Multiples	Pre-fixes	Sym-bols	Pro-nunciation
$1\ 000\ 000\ 000\ 000 = 10^{12}$	tera*	T	ter'a
$1\ 000\ 000\ 000 = 10^9$	giga*	G	ji'ga
$1\ 000\ 000 = 10^6$	mega	M	
$1\ 000 = 10^3$	kilo	k	
$100 = 10^2$	hecto	h	
$10 = 10^1$	deka	dk	
$0.1 = 10^{-1}$	deci	d	
$0.01 = 10^{-2}$	centi	c	
$0.001 = 10^{-3}$	milli	m	
$0.000\ 001 = 10^{-6}$	micro	μ	
$0.000\ 000\ 001 = 10^{-9}$	nano*	n	na'no
$0.000\ 000\ 000\ 001 = 10^{-12}$	pico*	p	pi'co

## Government Cuts Prices

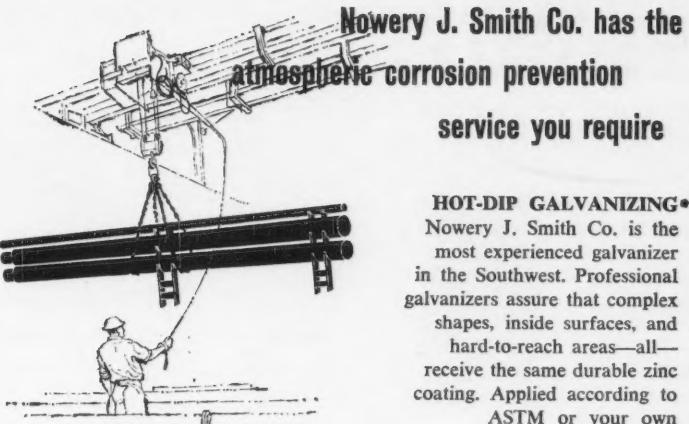
### 90 Percent on Helium-3

A 90 percent reduction in the price of helium-3, from \$1.50 to 15 cents per cubic centimeter, became effective January 1, according to the Atomic Energy Commission. Helium-3, packaged and distributed by the AEC by Oak Ridge National Laboratory, contains more protons per neutron than any other isotope and therefore forms a unique tool for producing nuclear reactions, permitting studies in which the capture of the two protons or the capture of a proton and a neutron can be directly compared.

## Inventions Conference

Inventors will have an opportunity to present their latest creations before manufacturers and investors at the National Inventions Conference and Exhibition set for June 20-24 at the Cleveland Engineering and Scientific Center, Cleveland, Ohio. The conference is sponsored by the Cleveland Engineering Society.

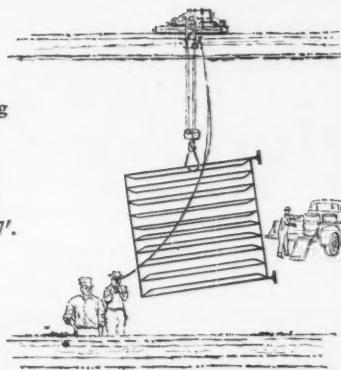
7464 copies of NACE Technical Committee Reports published in CORROSION were sold in 1958.



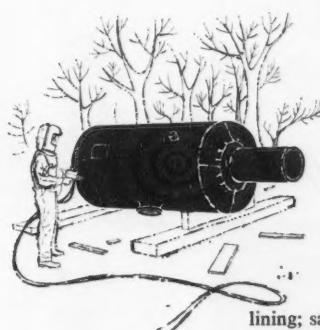
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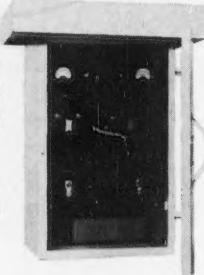
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ALUMINUM BOATS are being built to take advantage of the material's light weight and resistance to corrosion. The 45-foot aluminum hulled catamaran on the left has a top speed of 20 knots and will be used for sports fishing, passenger service and special purposes. The 35-foot aluminum pilot boat on the right has only a 3-foot draft and weighs 19,700 pounds.

## April 14 Corrosion Seminar Scheduled by ASM Chapter

A seminar on the mechanisms of corrosion is scheduled for April 14 by the Chicago-Western Chapter of the American Society for Metals to be held at the Argonne National Laboratory, Lemont, Illinois. Registration fee for the one-day seminar will be \$12.50, including a luncheon.

Speakers and their topics for the seminar are as follows: E. A. Gulbransen, Oxidation of Metals; H. T. Francis, Aqueous Corrosion; J. R. Weeks, Mechanisms of Liquid Metal Attack; J. H. De Van, Fused Salt Corrosion; and F. L. LaQue, Specific Corrosion Problems and Their Practical Solutions.

More than 30,000 franchised retail dealers sell motor trucks in the United States.

## 2 Divisional Meetings Set By AIME for April - June

Two divisional meetings of AIME are scheduled for April and June. On April 21-22, the Southwest Metals and Minerals Conference of AIME will be held at the Ambassador Hotel, Los Angeles, Cal. The program will emphasize papers on energy sources for fuel materials for the space age, special minerals for ground installations, a symposium on boron and other related data.

The 1960 International Conference on Powder Metallurgy will be held June 13-15 at the Biltmore Hotel, New York City. The conference will be co-sponsored by the AIME Powder Metallurgy Committee and the Metal Powder Industries Federation.

## Concrete Institute Builds New Chicago Headquarters

The Prestressed Concrete Institute has established new headquarters at 205 West Wacker Drive in Chicago, Ill. The move from Florida was designed to provide greater centralization of activity and dissemination of information to the building industry.

An up-to-date library will be maintained at the new headquarters for use of members, engineers, construction experts, architects, trade journal editors and students. It also will serve as liaison for exchange of information, research and new methods between members.

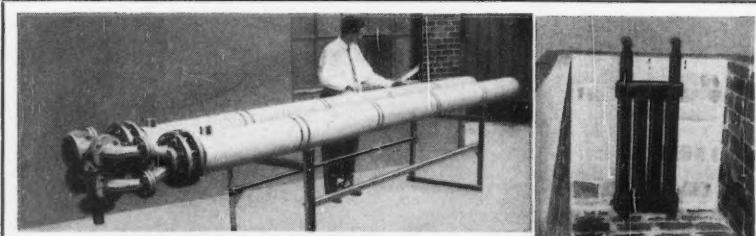
## Five Meetings Scheduled At Inco's Kure Beach

Five meetings have been scheduled at International Nickel Company's Kure Beach-Harbor Island Test Station, N. C.

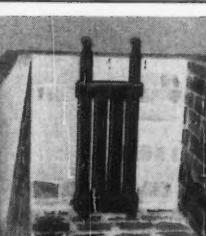
May 3-5: Chemical Engineers  
May 17-19: Power (Nuclear and Conventional Engineers)

May 31-June 3: Sea Horse Institute  
Sept. 13-15: Highway Engineers  
Oct. 11-13: Marine Engineers and Naval Architects.

8529 copies of NACE Technical Committee Reports published in CORROSION were sold in 1957.



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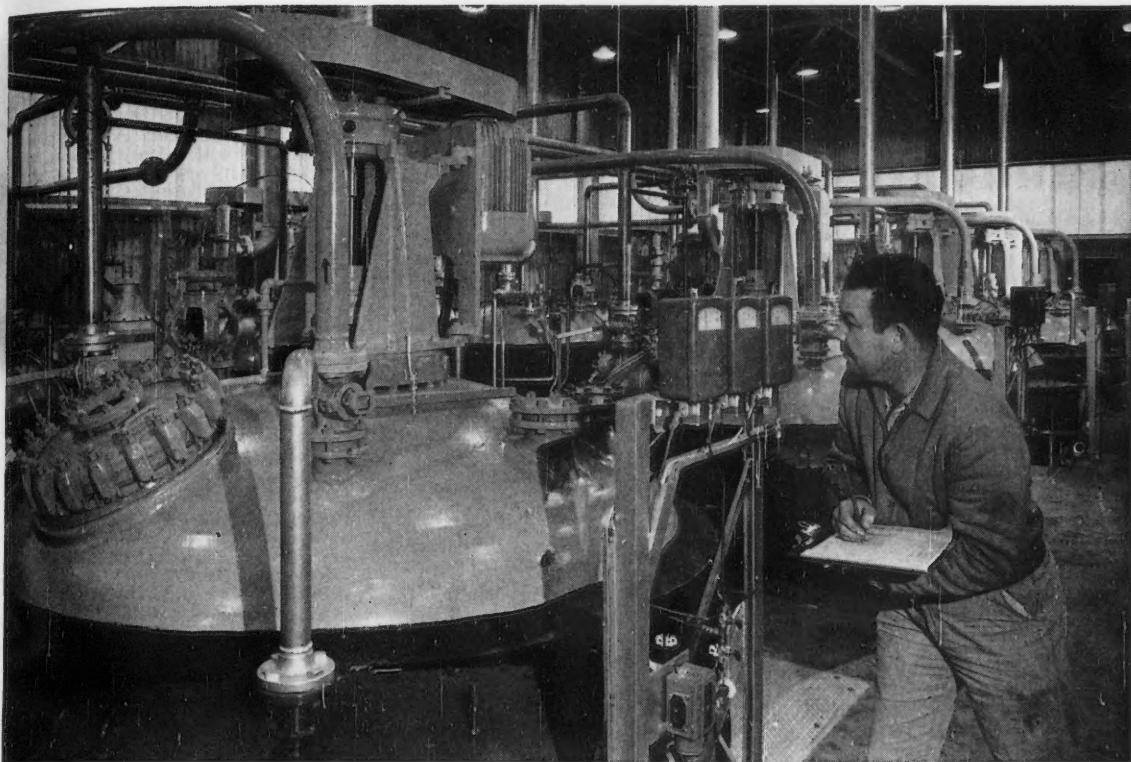
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## BOOK NEWS

**Gas Tungsten-Arc Welding of Titanium Piping and Tubing.** 16 pages, 6 x 9 inches, paper. 1959. The American Welding Society, 33 West 39th St., New York 18, N. Y. Per copy, \$1.50. A report of AWS committee on piping and tubing. Contents include process, power supply, tungsten electrodes, filler metal, titanium grades, joint design, cleaning, joint preparation, gas shielding, welding technique, heat treatment, weld quality tests.

Operations discussed are clearly illustrated.

**Physical Metallurgy.** By Bruce Chalmers. 468 pages, 5 1/2 x 9 inches, cloth. Dec. 9, 1959. John Wiley & Sons, Inc., 440 Fourth Ave., New York 16, N. Y. Per copy, \$12.50.

An outline of the modern concepts about the behavior of metals and alloys intended as a first textbook for physical metallurgy students or for metallurgists whose training was in the old concepts. The approach is qualitative with respect to development of physical concepts that can be applied to real cases.

Contents include: Structure of the atom, aggregates of atoms, structure-insensitive properties, imperfections in crystals, structure-sensitive properties, change of state; deformations, radiation damage and recovery processes; solid-state transformations. Discussion of corrosion phenomena is very limited.

There is an alphabetical subject index and an appendix of problems for students.

**Cathodic Protection.** By Lindsay M. Applegate. 229 pages, 6 x 9 inches, cloth. January 4, 1960. McGraw-Hill Book Company, Inc., 330 West 42nd St., New York 36, N. Y. Per copy, \$9. Developed from material prepared originally for corrosion and cathodic protection instruction of military personnel. The "do-it-yourself" approach is emphasized. Recognized published authorities have been used as sources supplemented by practical examples accumulated by the author during his work from Okinawa to North Africa and from Maine to California.

Chapters are headed: Corrosion and cathodic protection, instruments and measurements, surveys and current tests, coatings, anodes; design, construction and maintenance; protection of structures in contact with soil and protection under water and special conditions.

The book is extensively illustrated with diagrams and halftones. There is an alphabetical subject index.

**Physical Metallurgy of Stress Corrosion Fracture.** Thor N. Rhodin, editor. 394 pages, 6 x 9, cloth. 1959. Interscience Publishers, Inc., 250 Fifth Ave., New York 1, N. Y. and Interscience Publishers, Ltd., 88/90 Chancery Lane, London W.C. 2. Per copy \$13.

One of a series of volumes published by the Metallurgical Society of the American Institute of Mining, Metallurgical and Petroleum Engineers. The book consists of papers presented at a symposium at Pittsburgh April 2-3, 1959 with several organizations cooperating, including the National Association of Corrosion Engineers.

Contents include New Perspective on the Stress Corrosion Problem, by Herbert H. Uhlig; Effect of Electric Strain on the Electrode Potential of Metals by Ling Yang, G. T. Horne and G. M. Pound; Surface Structure and Chemical Interaction, Gert Ehrlich and D. Turnbull; Crack Propagation During Stress Corrosion, C. Edeleanu; Initiation and Propagation of Cracks in the Stress Corrosion of Alpha Brass and Similar Alloys, J. G. Hines and A. J. Forty;

Role of Corrosion Products in Crack Propagation in Austenitic Stainless Steel, Electron Microscope Studies, N. A. Nielsen; Effect of Stress and Environment on the Microtopology of the Corrosion Product, E. A. Gulbransen; Stress Corrosion of Single Crystals of Stainless Steel, H. W. Paxton, R. E. Reed, R. D. Leggett;

Metallographic and Crystallographic Examination of Stress Corrosion Cracks in Austenitic Cr-Ni Steels, J. G. Hines and R. W. Hull; Stress Corrosion Cracking of Austenitic Stainless Steels in High Temperature Chloride Waters, W. W. Kirk, F. H. Beck and M. G. Fontana; Effect of Composition on Stress Corrosion Cracking of Some Alloys Containing Nickel, H. R. Copson;

Dependence of Stress Corrosion Cracking Susceptibility on Age Hardening in a Copper-Nickel-Silicon Alloy, W. D. Robertson, E. G. Grenier, W. H. Davenport, V. F. Nole; Stress Corrosion Cracking in Low Carbon Steel, Hugh L. Logan; Phenomenon of Cracking and Fracture of Steel in the Presence of Hydrogen Corrosion Under Stress in the Presence of Moist Hydrogen Sulfide, Paul G. Bastien; Electrochemical Mechanism of Stress Corrosion Cracking of Mild Steel, H. J. Engell and A. Baumel; Current Problems for Additional Consideration, L. R. Scharfstein.

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**Cathodic Protection.** By J. H. Morgan. 325 pages 6 x 9 $\frac{1}{4}$  inches, cloth. 1960. The Macmillan Co., 60 Fifth Ave., New York 11, N.Y. Per copy \$12.

A thorough exposition of the whole field of cathodic protection with emphasis on practical applications. The author has made an exhaustive study of the literature on which the practice of cathodic protection engineering is based and has included at the end of each chapter references to the source of his information. A large percentage of the references are to articles published in CORROSION.

The book is liberally illustrated with both line and halftone plates, has numerous tables of data and is arranged in a logical sequence. A subject index is included. The style of the writing is clear and concise and uncluttered with technical obscurities. British money criteria are used when economic facts are considered.

The chapter headings are: Electrochemistry, corrosion and cathodic protection; practical cathodic protection parameters, resistivity and electrode resistance, sacrificial anodes, impressed current cathodic protection, cathodic protection of buried structures, cathodic protection in sea water, cathodic protection of structures containing electrolytes, stray current and interference corrosion, instruments for cathodic protection, cathodic protection economics.

**ASTM Standards on Copper and Copper Alloys.** 704 pages, 6 x 9 inches, cloth. December, 1959. American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa. Per copy \$7.50.

A compilation of ASTM standards, specifications and test methods on copper and copper alloys, cast and wrought copper and copper-alloy electrical conductors, non-ferrous metals used in copper alloys. The book is sponsored by ASTM Committee B-5.

The book contains hundreds of specifications, methods and definitions. There are appendices concerning classification of wrought copper-base alloys, regulations governing committee B-5, personnel of the committee and representatives of the committee on other committees. There is an alphabetical subject index.

**Tungsten. Bibliography 1953-58.** Physical Properties and Phase Diagrams. 39 pages, 8 $\frac{1}{2}$  x 11 inches, paper. 1959. Sylvania Electric Products, Inc., Chemical and Metallurgical Division, Towanda, Pa. Per Copy \$1.

A compilation of 409 abstracts arranged alphabetically by first author's names. Fifteen of the abstracts pertain to corrosion. There is a subject index and 19 phase diagrams of various tungsten combinations with other metals.

**Bibliography on Filing.** Classification and Indexing Systems for Engineering Offices and Libraries. ESL Bibliography No. 14. 1960. 33 pages, 8 $\frac{1}{2}$  x 11, mimeographed, paper. Compiled by Engineering Societies Library, 29 West 39th St., New York 16, N.Y. Per Copy \$2.

One hundred and fifty-five abstracts of articles dealing with systems for the filing and classification of technical information. Two of the three systems organized for corrosion pertain to the NACE Abstract Filing System. A wide variety of generalized and specialized systems are considered, most of them prepared by working engineers.

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## Equipment Services

# NEW PRODUCTS

## Materials Literature

### Ceramics

Operating temperatures to 2300 F are withstood by Fiberfrax Ceramic Fiber, according to a new brochure available from Carborundum Company, Buffalo Ave., Niagara Falls, N. Y. Properties, available forms and current uses of the high temperature material are given. Uses for domestic oil burner linings, furnace linings and critical applications in missiles also are discussed.

Vitrified clay pipe plant is being built at Ocala, Fla., by United States Concrete Pipe Company, subsidiary of Pittsburgh Coke & Chemical Company.

### Coatings, Application Equipment

Designed to overcome previous temperature and environment restrictions of oxygen-acetylene and oxygen-hydrogen techniques, a Plasmatron spray system with a hand-held gun is described in a technical bulletin available from Plasma-dyne, 3839 South Main St., Santa Ana, Cal. The S-Series Plasmatron system permits melting or vaporizing of any element in the periodic chart for spray coating, according to the company. A 4-pound plasma head, coaxial water and power cable, modular construction and central controls are other features of the system.

High-speed towing of a portable 6-horsepower compressor for airless spray painting is possible with a trailer-compressor unit available from the DeVilbiss Company, 300 Phillips Ave., Toledo, Ohio. The unit delivers 15 cubic feet per

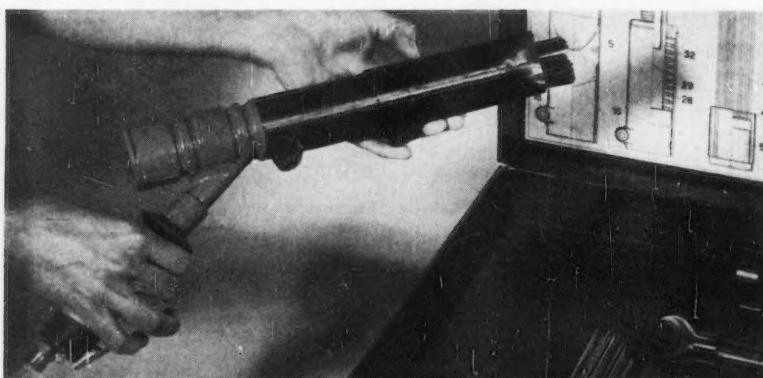
minute at 80 psi and 13.5 cubic feet per minute at 125 psi. The compressor has two cylinder, single stage pumps, forced air cooling, ball bearing construction, automatic, measured feed oiling and adjustments for normal or high pressure operation. The boat-type trailer has high speed wheel bearings so that the unit can be towed at highway speeds.

### Filters

Resistance to acids, alkalies, oxidizers and solvents (except a few chlorinated aromatics at high temperatures) has been designed into filter fabrics woven from polypropylene fiber, available from Technical Fabricators, Inc., 136 Washington Ave., Nutley, N. J. Polypropylene can be applied under hot (to 275 F) abrasive conditions and helps solve cake release problems on plate and frame presses, rotary drum and pressure leaf filters.

Effective trapping and removal of concentrated sulfuric acid sludge so that controlled volume pumps can work at peak efficiency are design features of a special sludge trap and filter manufactured by the Milton Roy Company, 1300 E. Mermaid Lane, Philadelphia 18, Pa. Bulletin 459 available from the manufacturer describes design features, operation and diagrams.

Greater heat resistance and better chemical resistance have been achieved in filtering fume dust by use of glass fabric filter bags. Longer bag life also has been experienced, according to Owens-Corning Fiberglas Corp., 717 Fifth Ave., New York, N. Y., supplier of the fabrics for manufacture of the bags being used for air pollution control.



**VON ARX AIR GUN** — unique new tool that excels in preparing uneven and inaccessible surfaces.

This versatile, light-weight tool is designed for really tough cleaning, de-scaling and de-rusting jobs. Labor saving, it pays for itself. Air-operated reciprocating needles adjust to contours automatically—easily and rapidly cleaning irregular surfaces such as rivets, crevices, grooves and corners. The VON ARX AIR GUN, in three sizes, comes in handy kit equipped with needles and chisels for specific applications. Representatives in principal cities. Write for descriptive brochure or demonstration.

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### Heaters

About 85 percent conversion of their energy into radiant heat similar to the sun's heat has been claimed for the General Electric T-3 Tubular Quartz Infrared Lamps. Used with an efficient reflector, the units are designed to make radiant heating practical for most indoor and outdoor applications. Units in 1-, 2- and 3-lamp fixtures ranging from 500 to 15,000 watt are available from Lumitor, Inc., 120 N. Peoria St., Chicago 7, Ill.

Drying, heating, baking and curing with 90 percent heat energy absorbed by the product is possible with the Vycor tubular heater and the Pyrex panel heater, according to a bulletin available from Corning Glass Works, Corning, N. Y. The Vycor unit emits infrared rays from wire coils enclosed in tubes of 96 percent silica glass. Radiant energy source in the Pyrex unit is a tempered borosilicate glass panel. One surface of the glass has been coated with an electrically conductive film that serves as a resistance element and heats the glass panel.

### Inhibitors

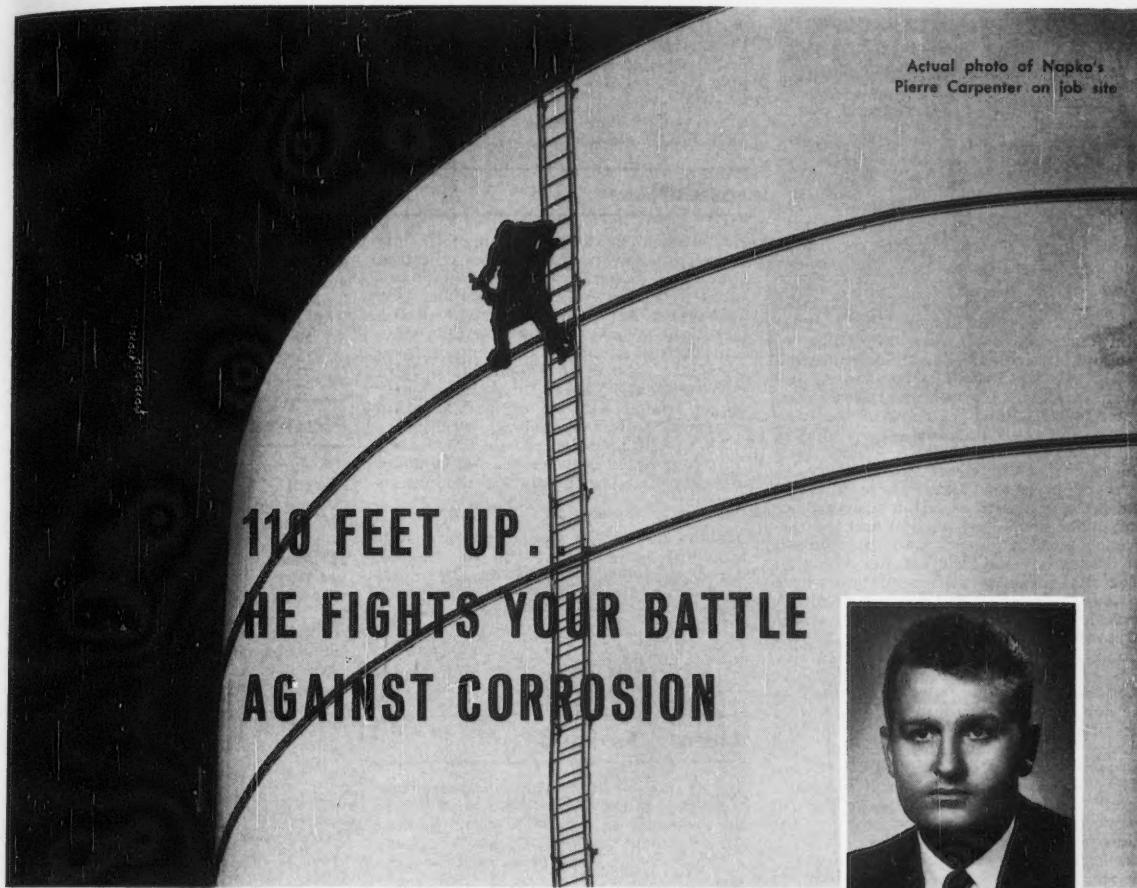
A protective film on all exposed metal surfaces including iron, steel, copper, copper alloys, aluminum, brass and solder is obtained by use of Corrosion Inhibitor CS, as described in a bulletin issued by Hagan Chemicals & Controls, Inc., Hagan Bldg., Pittsburgh 30, Pa. Compatible with solutions of alcohol and glycol-base antifreezes, the inhibitor is recommended for air conditioning systems, nuclear reactors, high temperature hot water systems, diesel and gas engines, compressors, process cooling water systems and ice melting systems.

Anionic, cationic and non-ionic classes of inhibitors are described by trade name, active ingredient, percent activity, physical state, general use, specific applications and properties in a catalog available from Onyx Oil & Chemical Co., 190 Warren, Jersey City, N. J.

### Instruments

A line operated portable pH meter that can be taken to the sample instead of the sample having to be brought to the meter has been designed by Analytical Measurements, Inc. 585 Main St., Chatham, N. J. Model 700 pH meter has a large scale, making readings possible within 0.02 pH, weighs five pounds and operates on any standard 115 a-c volt outlet.

Measurement of particulate matter as small as one-hundredth of a micron in diameter is claimed possible with a portable, high volume air sampler available from the Staplex Company, 777 Fifth Ave., Brooklyn, N. Y. Filter holder adapters from 6 x 9 inches to 12 x 12 inches are available so that large samplers (Continued on Page 74)



Actual photo of Napko's  
Pierre Carpenter on job site

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## NEW PRODUCTS

(Continued From Page 72)

ples of air can be collected in shorter times. A turbine type blower draws in large volumes of air. The sampler is being used to detect and measure smoke and smog, air hazards in mines, occupational dusts, factory health conditions and radioactive particles.

Metal losses as small as five billionths of an inch can be detected by Magnafilm corrosion indicators, according to the manufacturer, Crest Instruments, 11808 S. Bloomfield Ave., Santa Fe Springs, Cal., a division of Magna Products, Inc. Essential element of the indicators is a vacuum deposited film of metal, 2 to 50 millionths of an inch thick. Model 201 is used in small packages of precision parts where a visual indicator is sufficient. High contrast between the sensitized portion and the reference portion gives instant recognition of corrosive conditions. Model 301 is an electrical indicator for installation where visual indicators are not practical. A technical paper on these indicators was presented at the NACE 16th Annual Conference in Dallas, March 14-18.

Laminar flaws and corrosion loss can be detected by portable thickness testers described in Bulletin A-200 available from Branson Instruments, Inc., 40 Brown House Road, Stamford, Conn. These ultrasonic gages permit measurement of metal, glass and plastic by relating a variation in thickness to the change in resonant frequency. Model 5 uses transistors and weighs 4.75 pounds; Model 6 Audigage weighs 18 pounds and is designed for greater accuracy over a thickness range from 0.060 to 12.0 inches.

Designed to measure depth of scratches, cracks, blind holes, corrosion, dents and pits in all materials, the Ace Optical Micrometer also can measure height of spurs and protrusions. On transparent

materials, it can be used to measure thickness, depth of crazing, depth of fractures and depth and thickness of bubbles or seeds. Available in a variety of configurations, the instrument is manufactured by Air Cargo Equipment Co., 1121 East Colorado St., Glendale 5, Calif.

### Insulation

Recommended for insulating flexible connecting lines and for severe vibration conditions, Unarco Insutube, manufactured by Union Asbestos & Rubber Co., 1111 West Perry St., Bloomington, Ill., is a light weight, non-combustible flexible tubing designed for temperatures from 50 to 500 F. It is available with an uncoated outer jacket or with an outer jacket coated with an oil and abrasion resistant, self-extinguishing synthetic rubber. A heat-sealed plastic liner vapor barrier inserted between the outer jacket and fibrous glass insulating medium is available.

Molded fiber glass insulation for standard pipe fittings that can be stapled, wired or taped in place has been designed by Fibrous Glass Products, Inc., Alpa Plaza, Hicksville, Long Island, N. Y. It is available in four thicknesses from  $\frac{1}{2}$  to 2 inches in standard sizes for welded and cast ell, tees and special shapes.

### Metals, Ferrous

Ingots to 50 inches in diameter and weighing as much as 40,000 pounds will be produced at a new vacuum melting furnace plant to be built by Allegheny Ludlum Steel Corporation at its Waterbury Works in New York State. The 50-inch consumable electrode vacuum furnace will produce the high alloy super-metals and close specification low alloys required for supersonic aircraft, missiles, atomic reactors and other applications.

A new ultra high strength steel is being produced for the missile industry by

Universal-Cyclops Steel Corp., Bridgeville, Pa. When properly tempered to yield strength levels of 225,000 to 235,000 psi, the new alloy called Unimach UCX2 shows no susceptibility to notch sensitivity—one of the primary problems in the production of rocket motor cases. The alloy has shown excellent forming characteristics and good weldability for each fabrication, according to the manufacturer.

Approved methods of fabricating and welding titanium and titanium alloys are described in Booklet No. 3 available from Republic Steel, 1441 Republic Bldg., Cleveland 1, Ohio. Also available are Booklet No. 1 "Titanium Production and Use" and Booklet No. 2 "Titanium Grades and Properties."

### Plastics

Resistance to most acids, alkalies and solvents is claimed for a new epoxy floor developed by Kalman Floor Co., 110 East 42nd St., New York 17, N. Y. Designed to cure quickly and withstand abrasion, the floor formulation can be used to resurface worn industrial floors where speed is essential.

A new thermoplastic resin designed for high resistance to heat, light, corrosive chemicals and solvents has been developed by Pennsalt Chemicals Corporation, Three Penn Center, Philadelphia 2, Pa. The fluorine-containing resin called RC-2525 is stable under strong ultraviolet radiation and extreme weather conditions, according to the developer, and has high impact resistance.

Piping installation time is cut 25 to 50 percent when Fluoroflex-TS piping is used in high temperature and corrosive conditions, according to the manufacturer, Resistoflex Corporation, Roseland, N. J. This tetrafluoroethylene lined pipe and fittings have shown no attack by hydrofluoric, hydrochloric, sulfuric and nitric acids at temperatures to 500 F after being in service for over three years.

Texas district sales office for Union Carbide Plastics Company has been moved to 6300 North Central Expressway, Dallas 6, Texas. This office will handle sales of polyethylene, phenolic, vinyl, styrene and epoxy resins and compounds.

Resistance to corrosive fumes from nitric acid, hydrofluoric acid and dilute sulfuric acid at 180 F has been included in the design of a 46-foot ventilating hood built of reinforced plastics by the Ceilcote Company, 4832 Ridge Road, Cleveland 9, Ohio. The unit is a combination hood-and-duct designed for service in a large nickel processing plant. The hood is equipped with an internal washout spray pipe to eliminate collections of dust, scale and similar corrosive substances.

### Rubber

The first man-made rubber with the same molecular structure as natural tree rubber is being studied for commercial production by Goodrich-Gulf Chemicals, Inc., 3121 Euclid Ave., Cleveland 15, Ohio. Another new synthetic rubber called Ameripol CB (cis-poly-butadiene) also is being studied for production. It

(Continued on Page 76)

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# HOW CAUSTIC SODA IS KEPT IRON-FREE AT 285° F.

You are a producer of caustic soda faced with an unusual corrosion problem in which corrosive damage to equipment is not an important consideration: how can you economically protect your product from iron contamination from the steel tank cars that transport it?

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HYPALON lining has retained its original smooth finish.

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For more information send for a copy of PROTECTIVE LININGS AND COATINGS, a brochure designed for the engineer with a corrosion problem. For your copy write: E. I. du Pont de Nemours & Co. (Inc.), Elastomer Chemicals Department C-4, Wilmington 98, Delaware.



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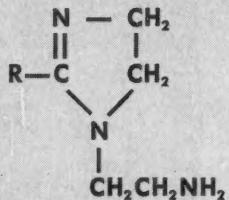
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Nalcamine G-39M has been designed expressly for use in products where pharmaceutical purity and color are not required. Thus it offers you all these important properties for a top corrosion inhibitor at a really *down to earth cost!*

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Nalcamine G-39M is a mixture of diamino imidazolines, consisting predominantly of the diamine shown above where the mixed alkyl chains are heptadecenyl and heptadecadienyl.

### TYPICAL ANALYSIS

Apparent Molecular Wt.	422
Apparent Combining Wt.	211
% Imidazoline . . .	94.0
% Titratable . . .	99.2
Spec. Gravity at 60°F . . .	0.948
Pour Point °F . . .	25
Viscosity at 60°F. cps . . .	334
Density—Lbs. per gallon . . .	7.91
Color . . .	Black

### SHIPPING

Nalcamine G-39M is shipped in fifty-four gallon, bung, unlined, non-returnable steel drums. Weight of each drum is approximately 420 lbs.

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Nalcamine G-39M can be handled or stored in iron or mild steel. It should be stored in a closed container.

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## NEW PRODUCTS

(Continued From Page 74)

is a stronger, tougher rubber at high temperatures than other general purpose synthetic rubbers, according to Goodrich-Gulf.

Application of natural and synthetic sheet rubber linings and plastic coatings are being handled at a new plant of Trevor Boyce Associates, Inc., P. O. Box 10171. The plant is at Dayton, Texas. This company has been appointed applicator for the B. F. Goodrich Rubber Company in the Gulf Coast area.

### Surface Preparation

A new chemical process that is claimed to eliminate all forms of electro polishing, machine and hand buffing has been developed by Chemclean Products Corp., 15-08 121 Street, College Point 56, N. Y. Called Ox-Off Lustre Dip, it is designed to leave a polished, smut free surface on 300 series stainless steel.

Sandblasting of interiors of pipelines in the ground has been developed by Klean-Kote, Inc., P. O. Box 488 LaPorte, Texas. It is designed to provide a perfect metal surface for lining pipeline walls with coatings such as epoxies, phenolics and polyesters. Sand suspended in a high velocity air stream is injected at a pig trap, fitting or special cut in the line and is exhausted at the other end, open to the atmosphere.

## MEN in the NEWS

Thomas E. Catlow, member of NACE and AIME, has been appointed sales coordinator for Associated Pipe Inspectors, Inc., Box 22302, Houston, Texas. He will work with the company's offices throughout Louisiana and Texas.

Harold R. Huntley has retired as chief engineer for American Telephone & Telegraph Co., New York, N.Y. He has been a member of NACE since 1946. He has been with the company since 1917.

John W. Baggott has been appointed chief process engineer for the Paint and Brush Division of Pittsburgh Plate Glass Co., Pittsburgh 22, Pa.

Fred W. Beitner, NACE member, has been appointed general sales manager of the Trent Tube Company, East Troy, Wis.

Richard H. Blair has been appointed superintendent of engineering and maintenance for Columbia-Southern Chemical Corporation's plant at Natrium, W. Va. William M. Halicks has been named general plant maintenance foreman. Both positions were recently created.

Henry C. Willms has been promoted to manager of Reynolds Metals Company's reduction plant at Jones Mills, Ark. He succeeds William E. Campbell, who was promoted to assistant manager of the company's reduction plant at Troutdale, Ore.

April, 1960

## RECORD AND REPORT

77

NACE member Lewis G. von Lossberg, consulting engineer with Sheppard T. Powell and Associates, 330 North Charles St., Baltimore 1, Md., presented a paper titled "Water Treatment in Paper Mill Power Plants" (co-authored by Hilary E. Bacon) at the 45th Annual Meeting of the Technical Association of the Pulp and Paper Industry held February 22-25 in New York City.

G. J. Weis has been transferred as a technical sales representative for Union Carbide Plastics Company to cover the South Atlantic region. His headquarters will be 57 Forsyth St., Atlanta 3, Ga.

T. J. Weisbruch will be manager of the new Indiana-Michigan district office recently opened at 3001 Fairfield Ave., Fort Wayne, Ind., by Dearborn Chemical Company.

Harry White has been appointed sales engineer for Pipe Inspectors of Houston, Texas.

Fred B. O'Mara has been named director of manufacturing for National Carbon Company, division of Union Carbide Corp., 100 East 42nd St., New York 17, N.Y.

NACE member Edwin A. Roman has been appointed general manager of H & H Sales and Engineering Co., 2111 Husson St., Houston 23, Texas.

Alexander Ross has been appointed as organic group leader, Research and Development Division of the Metal and Thermit Corp., 100 Park Ave., New York City.

(Continued on Page 78)

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## MEN in the NEWS

(Continued From Page 77)

**Arthur J. Wiltshire** has been appointed chief project engineer by Structural Fibers, Inc., Chardon, Ohio.

**Robinson F. Barker** and **James F. Junge** have been elected members of the Pittsburgh Plate Glass Company's board of directors. Mr. Barker, vice president and general manager of the glass division, has been with the company since 1935. Mr. Junge is director of investment research and a member of the board of directors of the Pitcairn Company.

**H. M. Rivers** has been appointed assistant director of Hall Laboratories, a di-

vision of Hagan Chemicals & Controls, Inc., Hagan Bldg., Pittsburgh 30, Pa.

**Fred D. Sides** is sales representative for Victaulic Company of America in the Panhandle and West Texas area. His headquarters will be the Victaulic Warehouse and office in Odessa, Texas.

**Stanton V. Sheppard** has been appointed manager of the new Process Equipment Department recently formed by the Ceilote Company, 4832 Ridge Road, Cleveland 9, Ohio.

**Norman J. Smith** has been named chief engineer of the instrument division of J. E. Lonergan Company, 211 Race St., Philadelphia 6, Pa.

**E. M. Turner** has been appointed district

sales representative for the recently opened sales office in Birmingham, Ala., by Alloy Steel Products Co., Linden, N.J.

**Norbert J. Connors** has been promoted to sales manager of the Pittsburgh district for the A. M. Byers Company, Clark Building, Pittsburgh, Pa. **Morton M. Jenkins** was promoted to assistant to the steel sales manager.

**Robert B. McKee** is the new vice president in charge of sales for the Aluminum Company of America, succeeding **Ralph V. Davies**, who retired recently. **F. J. Close** has been appointed to the new post of vice president and general sales manager. **William S. McChesney** has been appointed to the new position of general manager of market research and planning. **Samuel J. Simmons, Jr.**, will become general manager of the sales development divisions and industry sales.

**Wayne E. Glenn**, manager of Continental Oil Company's production department at Houston, Texas, is the new president of the Society of Petroleum Engineers. He succeeds **John S. Bell** of Humble Oil & Refining Co., Houston.

**Gabriel F. Doria** has been appointed technical representative for U. S. Stoneware Company. He will work out of the company's New York City offices at 60 East 42nd Street.

**Richard J. Grant** has been promoted to senior chemist in the physical chemical research group of Pittsburgh Coke & Chemical Company's research and development department. **John E. Urbanic** has joined the research and development department as chemist in the activated carbon research group.

**George C. Harrison, Jr.**, is now working on protective coatings for steel in the Metalworking Chemicals Division of Amchem Products, Inc., Ambler, Pa. Formerly, he was associated with Pennsalt Chemicals Corp., Philadelphia.

**Leslie B. Hartnett** has been appointed general manager of the Patterson-Sargent Company, St. Clair, Kopp and 38th Streets, Cleveland, Ohio, a subsidiary of H. K. Porter Company, Inc., Pittsburgh 19, Pa.

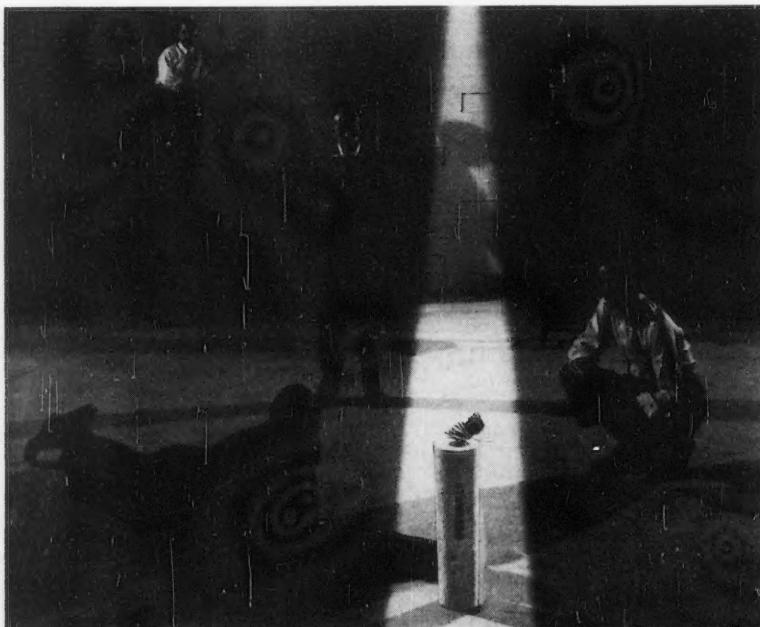
**Walter R. Hibbard, Jr.**, has been appointed manager of the metallurgy and ceramics research department at the General Electric Research Laboratory, Schenectady, N.Y.

**J. R. Johnstone** has been appointed administrative assistant in the office of William H. Feathers, president of National Carbon Company, division of Union Carbide Corp., 100 East 42nd St., New York 17, N.Y.

**D. K. Kauffman** has been appointed manager of manufacturing for the Ceilote Company, 4832 Ridge Road, Cleveland 9, Ohio.

**Eric R. Morgan** has been appointed director of research for Jones & Laughlin Steel Corp., 3 Gateway Center, Pittsburgh 30, Pa.

**William Moorehouse** has been promoted to senior engineer at the Akron, Ohio, chemical plant of the B. F. Goodrich Chemical Company.



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# CORROSION ABSTRACTS

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## CORROSION ABSTRACTS

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### 3. CHARACTERISTIC CORROSION PHENOMENA

#### 3.3 Biological Effects

322, 6.2.5, 2.3.5, 3.7.3  
**Intergranular Corrosion Testing by Polarographic Method.** (In Japanese.) Kazuyoshi Okano, Yukinori Ota, and Yoshiharu Kitamura. Corrosion Engineering (Japan), 8, No. 1, 5-6 (1959) Jan.

Bath voltage-ampere curves of various stainless steels in boiling sulfuric acid-copper sulfate solution was measured by a polarographic method. A relationship was found between intergranular corrosion, type of stainless steel and its heat treated condition. 18505

322, 6.3.6, 3.7.3  
**An Investigation of Hydrogen Embrittlement in Copper.** Einar Mattson and Franz Schuckher, AB Svenska Metallverken, Västerås, Sweden. J. Inst. Metals, 87, Pt. 8, 241247 (1959) April.

Deoxidation and the hydrogen embrittlement of oxygen-bearing copper have been investigated by means of annealing experiments in the range of 400 to 700 C. The effects of hydrogen pressure, annealing temperature, and annealing time were studied. At the beginning of the annealing period, deoxidation unaccompanied by cracking takes place through diffusion of oxygen to the surface (activation energy approximately 42 kcal/g-atom). After this incubation period deoxidation combined with cracking occurs as a consequence of penetra-

tion of hydrogen into the metal. Thus, the results quantitatively confirm the hypothesis proposed by Ransley (J. Inst. Metals, 65, 147, 1939). The rate-determining step for the hydrogen penetration seems to be either the transfer of hydrogen atoms from the surface into the interior or the diffusion of these atoms in the metal. Industrial bright annealing must be carried out within the incubation period. Maximum values of annealing temperature and hydrogen concentration have been determined for an incubation period of 1.5 hour. (auth)—NSA. 18449

#### 3.2.2

**Ubiquity of Localized Corrosion.** R. B. Mears. U. S. Steel Corp. J. Electrochem. Soc., 106, No. 5, 467-468 (1959) May.

It now appears that all commercially available metals contain dislocations and that, under appropriate conditions, localized corrosion (etch pits) can develop at dislocation sites. Very specific etchants must be used to reveal dislocation sites. One localized attack starts at any given area, electrochemical effects sustain it at that area, making it less probable that adjacent areas will corrode.—INCO. 18432

#### 3.2.2, 6.2.5, 2.3.4, 4.3.6

**Pitting Corrosion of Austenitic High-Alloy Chromium-Nickel Steels and Ferritic Chromium Steels in Aqueous Halide Solutions.** (In German.) Guenther Pier and Wilhelm Schwenk. Werkstoffe u. Korrosion, 10, 78-81 (1959) Feb.

Alloy steels (18 chromium, 10 nickel; 18 chromium, 10 nickel, 2 molybdenum; 18 chromium, 10 nickel, titanium stabilized; 18 chromium, 10 nickel, 2 molybdenum, titanium stabilized) investigated in ferric chloride, sodium chloride, sulfuric acid and potassium nitrate by the potassium ferri-cyanide test. Pitting appears as active-passive reaction.—RML. 18492

#### 3.2.2, 4.3.7

**Effect of Corrosion Produced by Aqueous Solutions of Hydrogen Sulfide on the Stress-Corrosion Cracking of Steel.** (In German.) Friedrich Karl Naumann and Wolfhard Carius. Arch. Eisenhüttenw., 30, 283-292 (1959) May.

Atomic hydrogen separates during cathodic reaction. This separation is not caused by ions discharge but results directly from the reduction of dissolved hydrogen sulfide molecules. 19 references.—RML. 18542

#### 3.2.3

**Adherence of Scale on Steel.** (In German.) Friedrich-Karl Peters and Hans Juergen Engell. Arch. Eisenhüttenw., 30, 275-282 (1959) May.

Effect of scale thickness, chemical composition of steel and oxidizing temperature on the adhesive strength. Increase in the thickness of scale layer is accompanied by a decrease in the adhesive strength. 21 references.—RML. 18528

#### 3.2.3, 6.2.2, 3.8.4

**High-Temperature Sulfitidation of Iron Alloys in Hydrogen Sulfide-Hydrogen Mixtures.** E. W. Haycock. Shell Develop-

opment Co. J. Electrochem. Soc., 106, No. 9, 764-771 (1959) Sept.

Rates of sulfide scale formation on pure iron and a series of chromium steels have been measured over the temperature range 400-550 C in hydrogen sulfide-hydrogen mixtures at a total pressure of 600 psi with a hydrogen-sulfide partial pressure of 0.6 psi for exposure periods up to 500 hr. A mechanism of scaling, involving the depletion of a barrier scale layer by recrystallization and grain growth processes, is proposed and substantiated by laboratory studies of the structural properties and mode of formation of scales grown on the various materials studied. A single kinetic equation, based on this mechanism, is shown to satisfy the rate curves over the complete range of conditions studied. A solid defect structure, protons trapped at metal ion vacancies, is suggested to explain the effect of hydrogen on the scaling process. The term "E" center is suggested to describe such a lattice irregularity. 18453

#### 3.2.3, 3.5.9, 6.3.6

**Oxide Films Formed at High Temperature on Copper Alloy Condenser Tubes.** (In Japanese.) Takemichi Otsu and Shiro Sato. Sumitomo Metals, 10, 167-173 (1958) July.

Pure copper, 70-30 brass, Admiralty brass, Albrac, 10% cupronickel and 30% cupro-nickel, heated at 400 to about 700 C in an electric furnace.—RML. 17524

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## 3.2.2, 6.4.2, 4.3.6

**Pitting Corrosion of Aluminium in Sodium Chloride Solutions.** T. Hagyard and J. R. Santhiappillai. *J. Applied Chem.*, **9**, Pt. 6, 323-330 (1959) June.

Anodic corrosion process in an artificial pit in aluminum undergoing active corrosion in dilute sodium chloride was examined by pH and potential measurements and by Schlieren observations. Evidence is produced that corrosion continues and spreads mainly in a downwards direction due to special ability of aluminum chloride solutions to activate passive aluminum surfaces, thus extending area of active metal along flow path of this solution. Anodic solution can dissolve large amounts of aluminum hydroxide yet still exhibit this behavior. Insoluble anodic products, postulated by previous workers, would seem to occur in a minor degree, if at all, inside of pit itself; any anodic hydrogen evolution would probably produce a basic aluminum chloride solution, not a mix-

ture of hydroxide and stoichiometric chloride. Photomicrographs, graphs, 15 references.—INCO. 18353

## 3.2.2, 6.2.5, 2.3.5

**Pitting Corrosion of Austenitic Chromium-Nickel Steels.** (In German.) H. Graefen. *Metalloberfläche*, **13**, 161-166 (1959) June.

Boiling experiments, measurement of potential-time and amperage-voltage curves. Development of pitting corrosion and its relationship to stress and intergranular corrosion. 10 references.—RML. 18368

## 3.2.2, 3.5.8, 6.3.20

**Observations on Microcrack Formation in Hydrogen-Embrittled Zirconium.** A. P. Young and C. M. Schwartz. Battelle Memorial Inst. Trans. Met. Soc. AIME, **212**, 309-310 (1958) June.

To understand the relationship of the hydride precipitate to the strain rate effect in tensile samples, the surfaces of chemically polished zirconium tensile samples strained at a slow rate and at an impact rate were examined in optical and electron microscopes. The apparent difference between slow-strain and impact tensile samples was the preponderance of twinning over slip in impact samples as compared to relatively little twinning in slow-strain samples. (auth) —NSA. 16697

## 3.2.3, 4.3.2, 6.3.20, 6.3.15

**Formation of Hydride Films on Titanium, Zirconium, Hafnium, Thorium Dissolving in Hydrofluoric Acid.** W. J. James and M. E. Straumanis. Univ. of Missouri. *J. Electrochem. Soc.*, **106**, No. 7, 631-632 (1959) July.

Small pieces of low hafnium content zirconium and titanium metal were immersed in 0.1 Normal hydrofluoric acid for 1 hour. The films were removed, dried and crushed to a powder. Powder diffraction pictures, using Cu K $\alpha$  radiation with a nickel filter, were taken, and "d" values of the pattern are tabulated and compared with data from many sources. Close agreement with hydride patterns and standards are indicated.—NSA. 18474

## 3.2.3, 6.3.10, 6.2.2

**Oxidation of Three Nickel-Iron Alloys and Iron at 800°C.** S. W. Kennedy, L. D. Calvert and M. Cohen. *Trans. Met. Soc., Amer. Inst. Mining Met. Eng.*, **215**, No. 1, 64-72 (1959).

Chemical analysis, weight-gain/time measurements, metallography and X-ray diffraction were used to study the composition and kinetics of formation of oxide scales on 99.95% iron and its binary alloys with 25.6, 75.4 and 84.2 weight-% nickel. The two high-nickel alloys form a three-layer scale, consisting of porous nickel monoxide next to the metal, a spinel phase which contains combined nickel in the case of the 84% nickel alloy, and  $\alpha$ -ferric oxide on the outside; the low-nickel alloy produces a scale of spinel containing traces of ferrous oxide and nickel monoxide near to the metal, and  $\alpha$ -ferric oxide; in most cases the spinel layer has the approximate formula  $Ni_{0.4}Fe_{2.8}O_4$  and the  $\alpha$ -ferric oxide whose thickness decreases with increasing nickel content has preferred orientation. Deep subscale formation occurs only in the low-nickel alloys and is associated with cracking of the scale layers and subsequent oxygen penetration along the grain boundaries; small amounts of subscale formation and intergranular oxidation occur in the other alloys. Although weight-gain/time curves

are not reproducible and have steps, X-ray data show that after a short initial period the thickness of the total scale, as well as that of the individual layers, increases according to a simple parabolic law. 11 references.—MA. 18469

## 3.2.3, 3.7.3

**Scale Formation on Hot Rolled Sheets.** (In German.) August Lueth, Arch. Eisenhüttenw., **30**, 71-78 (1959) Feb.

Scale on sheets in rolled conditions. Sheet scale behavior as affected by annealing in controlled atmosphere.—RML. 18412

## 3.2.3, 6.3.6, 3.4.7

**An Investigation of Chemical Variables Affecting the Formation of Films on Copper in Aqueous Solutions.** W. H. Davenport, V. F. Nole and W. D. Robertson. Chase Brass and Copper Co. *J. Electrochem. Soc.*, **106**, 1005-1009 (1959) Dec.

Growth of copper oxide films in aqueous solutions has been studied by an electrometric method. Quantitative data have been obtained on the growth of films as a function of the solution variables; anion type and concentration, pH, and oxygen concentration. In air-stirred chloride solutions, film thickness increases with increasing chloride concentration, passes through a maximum in solutions approximately 0.01M, and then decreases. A maximum also is obtained for pH dependence in the range pH 5.9-9.0. Cuprous oxide is formed below about pH 10.5 and cupric oxide predominates at higher values. For equimolar solutions (0.1M), film growth rate in sodium chloride solutions is about double that in sodium sulfate. In both cases the rate is linear and limited by oxygen availability under the conditions described. In high-purity water, in the presence of excess oxygen, the rate of growth is approximately parabolic, but the extremely thin films formed preclude detailed analysis with the present technique. 18311

## 3.3.1, 6.6.8

**Biological Deterioration of Plastics.** H. J. Hueck. *Plastica*, **12**, No. 1, 24-36 (1959).

Nature of the deterioration is considered both from the point of view of the type of damage, whether mechanical or chemical and from the point of view of the agent, whether microorganism, insect or rodent. Examples given include mildew of polyvinyl chloride coatings, development of bacteria in plastic water-pipes and damage to polyvinyl chloride insulated wiring by rodents. 23 references.—RPI. 18460

## 3.2.2, 6.3.15, 3.7.2

**Hydrogen Contamination in Titanium and Titanium Alloys. Part IV: The Effect of Hydrogen on the Mechanical Properties and Control of Hydrogen in Titanium Alloys.** Period covered: December 1954 to March 1957. D. N. Williams, F. R. Schwartzberg, P. R. Wilson, W. M. Albrecht, M. W. Mallott and R. I. Jaffee. Battelle Memorial Inst. U. S. Wright Air Development Center, Technical Report 54-616 (Pt. IV), June 7, 1957, 237 pp.

A comprehensive investigation of hydrogen in titanium alloys was carried out. Tests were conducted to gain an insight into the mechanism of the slow-strain embrittlement of titanium alloys by hydrogen, and a tentative theory is presented. Eighty titanium alloys were examined in the stabilized condition to determine the effect of composition on



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## CORROSION ABSTRACTS

the tendency toward hydrogen embrittlement. The effect of microstructural variations on the tendency toward embrittlement was also studied. In addition to studying the effects of hydrogen on the properties of titanium, factors affecting hydrogen pickup by titanium and methods of removing hydrogen from titanium were investigated. Included were investigations of low pressure solubility, degassing methods, and the pickup of hydrogen from various atmospheres.—NSA. 15159

## 3.2.3, 6.2.3

**Studies on the Structure and Adhesive Strength of Scale Layers on Unalloyed Steel Plates.** (In German.) Hans-Jürgen Engel and Friedrich-Karl Peters. Arch. Eisenhüttenw., 28, 567-574 (1957) Sept. Formation of roll scales and annealing scales; interrelationship between sealing, adhesive strength, and oxidation kinetics.—BTR. 14952

## 3.2.3, 7.6.8, 4.6.1, 5.7.9

**Study of Physical Antiscalting Treatments.** (In French.) J. Laureys and M. Pourbaix. Cebelcor, Rapport Technique No. 56, Sept., 1957, 36 pp.

Laboratory installation for evaluation of efficacy of water treatment methods in the case of distilling equipment. Effectiveness of magnetic and electromagnetic treatments, mercury floats, and inoculation with sodium metaphosphate and sodium uranate. Influence of a prior trickling treatment on scale-forming effect of nontreated water and magnetically treated water. Physicochemical effects connected with conditions of efficacy of physical treatments. Effect of external factors on crystallization phenomena. Current research program of Cebelcor Commission on Antiscaling Formation Processes. 9 references.—MR. 16223

## 3.2.3, 6.3.6

**Discontinuous Growth of Cuprous Oxide on Copper.** (In German.) E. Menzel, W. Stössel and C. Menzel-Kopp. Z. Naturforsch., Pt. A, 12, 404-409 (1957) May.

## 3.2.2, 6.3.6, 3.7.3

**An investigation of Hydrogen Embrittlement in Copper.** Einar Mattsson and Franz Schückher. J. Inst. Metals, 87, No. 8, 241-247 (1958-59).

Deoxidation and the hydrogen embrittlement of oxygen-bearing copper were investigated by means of annealing experiments in the range 400-700°C. The effects of hydrogen pressure, annealing temperature, and annealing time were studied. At the beginning of the annealing period, deoxidation unaccompanied by cracking takes place through diffusion of oxygen to the surface (activation energy ~ 42 kcal./g.-atom). After this incubation period deoxidation combined with cracking occurs as a consequence of penetration of hydrogen into the metal. Thus, the results quantitatively confirm the hypothesis proposed by Ransley (*ibid.*, 65, 147, 1939). The rate-determining step for the hydrogen penetration seems to be either the transfer of hydrogen atoms from the surface into the interior or the diffusion of these atoms in the metal. Industrial bright annealing must be carried out within the incubation period. Maximum values of annealing temperatures and hydrogen concentration were determined for an incubation period of 1.5 hours. (auth)—NSA. 18339

56, 118-119, 121, 123-124 (1958) Sept. 8; Chem. Abstracts, 53, No. 3, 2590b (1959) Feb. 10.

Filiform corrosion has been observed in a variety of alkanolamine systems. This type of corrosion can be studied in the laboratory by immersing a mild steel specimen in an aerated 1 percent diethanolamine solution containing 5 percent sodium chloride. The specimens are evaluated by making movies with a 3-min. time lapse over a 3-day period. Static and dynamic tests were made. Field studies are discussed. The only type of corrosion inhibitor found to be effective was a condensation product of polyethyleneamines and unsaturated alcohols.—RPI. 18198

## 3.2.3, 6.2.3, 3.7.4

**Structure of Scale on Plain Carbon Steels.** S. Garber. Nature, 183, No. 4672, 1387-1388 (1959) May 16.

It was generally assumed that structure of thin scales on products of modern continuous rod and strip mills conformed to classical picture of scale structure. However, during microscopic examination of scales of these products, unusual types of structure were observed (photomicrographs). One type is called "sandwich" structure. There is almost no evidence of blistering and it is essentially a continuous scale with parent metal. Innermost layer in contact with parent metal is magnetite. Middle-layer is wustite and next layer is again magnetite. In some instances a thin outer layer of hematite is also observed. In other structures observed, entire scale appears to be composed of a single phase of primary magnetite. Scale towards edges of strip also has a thin hematite layer on top of magnetite layer. Whereas decomposed wustite phase can be readily explained by existing knowledge, it is rather difficult to visualize mechanism of formation of inner magnetite layer next to parent metal. 5 references.—INCO. 18275

## 3.2.3, 6.3.9, 3.5.9

**Contribution to Oxide Formation on Molybdenum.** (In German.) H. J. Booss. Orsam G.M.B.H., Berlin. Metall., 13, 555-559 (1959) June.

Scaling of molybdenum sheets was investigated between 450 and 550°C. The preliminary treatment, such as annealing or etching, has a strong effect on the absolute value of the scaling constant. Both a structural resolution and a surface roughening increase the scaling constant. From 370 to 380 and from 460 to 470°C a structural transformation of the exterior oxide layer  $\text{MoO}_3$  occurs. The transformation is partly responsible for the pitting formation in the temperature range from 450 to 550°C. The activation energy of the scaling velocity in the range from 450 to 550°C is apparently only 46 kcal/mole. This value is affected by surface modifications and structural changes. (auth)—NSA. 18339

## 3.4 Chemical Effects

## 3.4.3, 8.4.5

**Product Purity and Corrosion in CPP Intercycle Evaporators.** W. J. Guy. American Cyanamid Co. U. S. Atomic Energy Commission Pubn., IDO-14216, Feb. 11, 1953 (Declassified February 27, 1957), 7 pp. Available from Office of Technical Services, Washington, D. C.

The contribution of impurities from corrosion is small. Impurities of alumin-

um and silicon are greater than expected. Part of the silicon is from the 10 ppm silicon in demineralized water. The use of distilled water would cut this down. Iron and sodium content are acceptably low. The corrosion rate in the product evaporator is 0.75 mil/yr and in the strip columns it is 1.5 mil/yr.—NSA. 16194



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T-5A-4 A Bibliography on Corrosion by Chlorine. A Report of Technical Unit Committee T-5A on Corrosion in the Chemical Manufacturing Industry. (Compiled by Task Group T-5A-4 on Chlorine.) Pub. 56-2, Per Copy \$1.50.

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## 3.4.3, 6.4.2, 3.4.8

**Observations on the Corrosion of Aluminum and Its Alloys in Relation to Chemical Examination of the Corrosion Products.** R. A. Hine. *J. Applied Chem.*, 9, Pt. 1, 43-49 (1959) Jan.

Contribution which analysis of products of corrosion can make to methods of investigating causes of corrosion of aluminum alloys in service, is described and illustrated with analyses of actual samples. Prevalence of corrosion involving either chloride or heavy metal ions is indicated by a summary of results of 130 case histories, outlining methods used to analyze corrosion products and discussing action of certain types of corrosion (e.g. galvanic). Individual contaminants covered include chloride, sulfate, nitrates, copper, iron, zinc, mercury, other metals (lead, tin), and other corrosive agents (e.g. water). An aggressive agent was detected in 100 cases (77%); of these, > 0.1 chloride was present together in 6 of above cases. Of remaining 11 classifiable cases, contact with iron or ferrous ferric oxide and exposure to alkaline or acid conditions were chief factors responsible. Flow-sheet diagram outlines methods of analysis; analyses of corrosion products are tabulated. 16 references.—INCO.

17717

## 3.4.3, 6.3.20

**Significance of White Corrosion Products or Zircaloy.** A. B. Riedinger. Knolls Atomic Power Lab. U. S. Atomic Energy Commission Pubn., KAPL-M-ABR-6, Sept. 4, 1958, 9 pp. Available from Office of Technical Services, Washington, D. C.

Corrosion products on Zircaloy are not desirable, but they are not necessarily indications of penetrating or catastrophic attack. The seriousness of the accelerated attack resulting in white corrosion products can frequently be determined by the appearance. The white corrosion products formed due to surface impurities have no effect on the structural strength of the Zircaloy but may seriously affect the heat-transfer characteristics or the amount of  $ZrO_2$  released to the stream.—NSA.

17579

## 3.4.3, 4.3.3

**Products of the Corrosion of Steel in Alkaline Sulfide Solutions.** (In English.) Eino Uusitalo. Suomen Kemistilehti, 31, No. 11, 367-371 (1958).

Iron and sulfur contents were found to vary between limits that correspond to the formulas  $Fe_2S_3$  and  $FeS_2$ . According to the X-ray patterns the corrosion products consisted of troilite ( $FeS$ ) or pyrrhotite ( $Fe_{0.85}S$ ), and pyrite ( $FeS_2$ ). The formation of kansite,  $Fe_2S_3$ , is also probable in the early stages of corrosion. 15 references.—RML.

17631

## 3.4.3, 2.3.7, 3.7.3, 6.4.1

**The Nature of Contact Corrosion Products Formed During Tension-Compression Tests on Riveted Joints in Light Alloys of the AG.5 Type.** (In French.) Mme A. R. Weill. *Rev. Mét.*, 55, No. 1, 61-66 (1958).

During tension-compression tests an abundant formation of powdery corrosion products was observed, containing, e.g., aluminum 46.6, magnesium 3.8%, and some nitrogen. X-ray-diffraction analysis revealed the presence of mono- and tri-hydrated forms of aluminum oxide. The black layer that surrounds

the rivets varies from matte to brilliant, suggesting that the degree of hydration varies. Anhydrous aluminum oxide appears to be absent. The damage caused to the surfaces by these products is discussed in terms of their hardness compared with that of the alloy.—MA.

17669

## 3.4.8, 3.8.3, 6.2.5

**Effect of Halogen Ions on the Anodic Passivation of 13 Chromium Stainless Steels.** (In Japanese.) Namio Ohtani and Hideo Sugawara. *J. Electrochem. Soc., Japan*, 26, No. 4-6, E-63-E-65 (1958).

Passivation dependent on  $X^-/OH^-$ ; rise of solution, differences on  $Cl^-$  and  $Br^-$  from that of  $F^-$  and  $I^-$ .—MR.

17528

## 3.4.8, 3.8.3, 6.3.15, 3.2.2

**Effect of Halogen Ions on the Anodic Passivation of Titanium.** (In Japanese.) S. Morioka and A. Umezono. *J. Japan Inst. Metal*, 23, No. 3, 185-189 (1959) March; See also: *Corrosion Engineering (Japan)*, 8, No. 8, 327-329 (1959) August.

Effect of halogen ions on the anodic passivation of titanium in acid, neutral and alkaline solutions has been studied. (1) In the presence of  $F^-$ , in an acid solution, severe attack of titanium occurs due to chemical dissolution of the oxide film by undissociated hydrofluoric acid. However, in neutral and alkaline solutions titanium is not attacked. (2)  $Cl^-$  does not prevent the passivation of titanium due to the very high overvoltage on titanium. (3)  $Br^-$  induces pitting corrosion in neutral solutions. (4)  $I^-$  shows high overvoltage in acid and neutral solutions but not in alkaline solution below the discharge potential of the hydroxyl ion. No pitting corrosion occurs by the discharge of  $I^-$ , since the discharging points tend to spread all-over and to disperse the electrolytic current. The corrosion resistivity of titanium in halogen-bearing solutions is due to its larger affinity to oxygen than to halogen. Therefore, in aqueous solutions the amount of dissolved oxygen, hydroxyl ion, or oxidizing agent has a significant effect on the corrosion resistivity of titanium. No pitting corrosion takes place unless a considerable external electrolytic current is applied, and thus, in natural environment, corrosion is usually prevented.—JSPS.

18443

## 3.4.8, 3.6.8, 3.8.2

**Effect of Specific Adsorption of Anions on Hydrogen Overvoltage.** Ya. M. Kolotyrkin. *Trans. Faraday Soc.*, 55, No. 3, 455-462 (1959).

$I^{2-}$  was used to measure the adsorption of  $I^-$  from aqueous solution on silver and lead electrodes. Measurements were made of the effect on the rate of hydrogen evolution of the adsorption of  $Cl^-$ ,  $Br^-$  and  $I^-$ . These results are discussed in terms of an effect on the potential within the double layer and an effect on the metal-hydrogen bond strength; cf. Randles and Somerton, *ibid.*, 48, 937, 951 (1952). 13 references.—MA.

18473

## 3.5 Physical and Mechanical Effects

## 3.5.3, 6.4.2

**Nature of the Abrasion Products of Aluminum Surfaces.** (In Italian.) M. Paganelli. *Alluminio*, 27, No. 10, 429-434 (1958).

The powder produced by friction between aluminum sheets was examined by X-ray diffraction, micrography and chemical analysis. It consists of tiny aluminum oxide-coated aluminum particles, formed by mechanical wear and abrasion. Its dark color is due to the small particle size.—MA.

17554

## 3.5.3

**Mechanism of Cavitation.** E. G. Richardson. *Wear*, 2, 97-106 (1958) Nov.

Measurements of the number and size of the gaseous nuclei which exist in water and other liquids and which give rise to cavitation and consequent erosion in hydraulic machinery. The nuclei may be removed by long standing or by application of pressure. Nuclei are detected by the absorption of an acoustic signal which they produce in a "reverberation vessel" containing the liquid. 4 references.—MR.

17586

## 3.5.3, 5.2.1

**Cavitation Damage. A Review of Present Knowledge.** D. J. Godfrey. Paper presented at Corrosion Group Meeting, Soc. Chem. Ind., October 22, 1958; *Chem. & Ind.*, No. 23, 686-691 (1959) June 6.

A review on cavitation covers the hydromechanical theory of cavitation erosion, the difference between cavitation erosion and impingement attack, the effect of cathodic protection and of entrained and dissolved air in reducing cavitation, environmental factors which affect cavitation erosion resistance; and properties of materials which affect cavitation erosion. 68 references.

18289

## 3.5.4, 8.4.5, 6.2.5

**Effects of Irradiation on the Type 347 Stainless Steel Flow Separator in the EBR-I Core.** R. E. Bailey and M. A. Silliman. Paper before American Society for Testing Materials, 61st Annual Meeting, Symposium on Radiation Effects on Materials, Boston, June 24, 1958. Am. Soc. Testing Materials, Special Technical Pubn. No. 233, 84-101; disc. 102 (1958). Available from the Society, 1916 Race Street, Philadelphia, Pennsylvania.

18433

Flow separator, made from 0.088-in. thick Type 347 hot-rolled sheet, was used to define flow paths of sodium potassium coolant in Mark-I and Mark-II cores of EBR-I. Test specimens were taken from region of maximum neutron exposure ( $2 \times 10^{24} \text{nvt}$ ) and from region of fairly uniform exposure ( $6.2 \times 10^{24} \text{nvt}$ ). Effect of irradiation on tensile properties, hardness, electrical resistivity and ferromagnetic properties were determined. Tests were made on as-received material and on annealed specimens to determine rates and temperatures at which radiation effects could be annealed out. Metallographic examination failed to reveal any changes in microstructure of irradiated and unirradiated material. Effects of irradiation—increased hardness and decreased ductility may be annealed out at temperatures as low as 500°C. Post-irradiation heat treatment promotes formation of heavy, uneven carbide at grain boundaries. Magnetic measurements show that fast neutron irradiation precipitates ferrites in Type 347. Tables, graphs, photomicrographs. 15 references.—INCO.

18077

Electrode corrosion. Corrosion, Soc., 106, 1958. Electrode corrosion is a reference to zinc, balsa wood, experiment, track corrosion and erosion current. purely magnetic. discreet process exists. It materials which can take place of a contact which can metal by removal of crating to form localized forming r

## 3.5.5, 5.3.4

**Nature, Cause and Effect of the Porosity in Electrodeposits. Pt. IV. Influence of Gas Bubbles on the Formation**

**of Pores.** AES Research Project No. 13. F. Ogburn and D. W. Ernst. National Bureau Standards. Plating, **46**, No. 8, 957-958 (1959) Aug.

One mechanism for formation of pore in electrodeposits involves inclusion of surface gas bubble. Whether pore becomes continuous or bridged depends upon relative rates of hydrogen discharge and nickel deposition. Cell is described which permits observations of simulated "cross section" of nickel deposit while it is being formed. Cell has concentric cylindrical electrode system and transparent Saran bottom. Cathode (center electrode) is pressed onto thin Saran bottom, and when placed on metallograph stage, plating taking place on periphery of cathode is observed. Series of photomicrographs show the enclosing of gas bubble. 7 references.—INCO. 18451

### 3.5.8, 6.2.5, 5.2.1

**Stress Corrosion Cracking of Austenitic Stainless Steel in Aqueous Magnesium Chloride Solution.** (In French.) T. P. Hoar and J. G. Hines. Métaux, Corrosion-Industries, **34**, 36-47 (1959) Jan.

Experiments on alloy steel wires under tension showed that corrosion cracking may be avoided by cathodic protection. Resume of author's studies since 1951 on fundamental mechanism of stress corrosion cracking. 23 references. 18464

### 3.5.8, 6.2.5, 5.9.1

**Study of Stress-Corrosion of 18-8 Type Austenitic Steels.** (In French.) Joseph Hochmann. Compt. rend., **248**, 2999-3000 (1959) May 25.

Study of influence of surface condition on corrosion resistance. Pickled and polished specimens broke after periods of time ranging from 2 to 24 hours. No cracking was noted in sand-blasted specimen after 150 hours.—RML. 18436

### 3.5.8, 1.5

**Internal Stresses and Fatigue in Metals.** G. M. Rassweiler and W. L. Grube, Editors. Book, 1959, 451 pp. Elsevier Publishing Co.

Proceedings of Symposium on Internal Stresses and Fatigue in Metals, held at General Motors Technical Center, Detroit and Warren, Michigan on September 4-5, 1958.—INCO. 18526

### 3.5.8, 3.2.2, 3.8.2, 6.4.4

**Electrochemical Aspects of Stress Corrosion.** D. K. Priest. J. Electrochem. Soc., **106**, No. 4, 358-360 (1959) April.

Electrochemical nature of stress corrosion is demonstrated with special reference to JI alloy (6% aluminum, 1% zinc, balance magnesium). In view of experimental evidence with regard to crack corrosion rates, cathodic protection and the nature of measured corrosion currents, it seems unlikely that purely mechanical cracking plays any discreet part in the stress corrosion process unless a distinctly brittle phase exists. It is concluded that in ductile materials transgranular stress corrosion takes place only under the driving force of a continuous electrochemical action which causes very localized removal of metal by corrosion. The site of metal removal (the crack tip) and the direction of cracking are determined by stress acting to cause localized potential differences through the interruption of film forming mechanisms or by intense, localized plastic deformation.—ALL. 18497

### 3.5.8, 6.2.5

**Resistance to Stress Corrosion of 12% Chromium Stainless Steel.** R. E. Lochen and E. R. Miller. Allis-Chalmers Research Labs. Ind. & Eng. Chem., **51**, No. 6, 763-764 (1959) June.

Stress corrosion tests of Type 403-0.05 molybdenum, Type 403-0.50 molybdenum, and Type 422 were made in 0.5% acetic acid saturated with hydrogen sulfide. Resistance to stress corrosion increased under following conditions: increased tempering temperature above 1000 F; addition of secondary alloying elements; decrease in applied stress; and vapor-blasted rather than an as-ground finish. Time to failure and impact strength increased slightly with an increase in tempering temperature up to 500 F. Above 500 F, time to failure and impact strength decreased to a minimum at about 800 F, but at still higher temperatures, both time to failure and impact strength increased (graph). Correlation also existed between hardness and time to failure at tempering temperatures above about 1000 F. Type 422 was more resistant than both heats of Type 403. Graphs, tables, 14 references.—INCO. 18407

## 3.6 Electrochemical Effects

### 3.6.6, 5.3.4, 3.7.2, 6.4.4, 4.2.5

**Galvanic Relationships Between Aluminum Alloys and Magnesium Alloys. Pt. II. Aluminum Cladding on Magnesium.** M. R. Bothwell. Dow Metal Products Co. J. Electrochem. Soc., **106**, 1019-1021 (1959) Dec.

Cladding of magnesium AZ31A alloy with 99.99 percent aluminum greatly improves its resistance to saline environments. The protective ability of the cladding is little changed by incorporation therein of magnesium, silicon or manganese, but it is impaired greatly by iron and copper and more mildly by zinc. The presence in seawater of soluble magnesium salts greatly diminishes the effects of iron, copper and zinc from that seen in sodium chloride solution. This decrease is probably due to decreased cathodic attack of the aluminum at the lower equilibrium pH of the seawater environment. 18218

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**Its Corrosion, and Antimony Electrodes.** Arthur L. Pitman, Marcel Pourbaix and Nina de Zoubov. *J. Electrochem. Soc.*, **104**, No. 10, 594-600 (1957) October.

Using methods and conventions described previously together with the available thermodynamic and electrochemical data, equations have been formulated and represented as a potential-pH equilibrium diagram of the antimony-water system for 25° C. Other diagrams are presented for the domains of solubility, predominance of ions, and for corrosion. The reason for the observed resistance of antimony coatings to hydrochloric and hydrofluoric acids is made clear. Applications are made to the antimony electrodes. The free energies of formation of the antimonate,  $Sb(OH)_6^-$ , and the  $SbO_2^+$  ion have been calculated and are given. 15399

### 3.6.5, 3.6.8, 3.8.2, 5.9.4

**The Science of Corrosion.** J. M. Kape. *Corrosion Prevention and Control*, **4**, No. 7, 37-41, 52 (1957).

The change in energy when metal ions pass into solution, its relation to the electrode potential which the metal takes up, and the change of this potential with time is discussed. The potential/pH diagram for iron dissolving in water is discussed. The use of a potentiostat in the detection of polarization curves is described and the importance of such curves in the prevention of corrosion is stressed with particular reference to the anodic oxidation of aluminum and other metals. 14 references.—MA. 15896

### 3.6.6

**Corrosion at Bimetallic Contacts.** (In Finnish.) Tenho Snek. *Teknillisen Keinian Aikakausilehti*, **14**, 137-144 (1957) March 30.

A review of recent investigations.—BTR. 14786

### 3.6.9, 5.2.1

**Stray-Current Corrosion of Underground Distribution Systems.** Donald W. Barron. *Albert F. Ganz, Inc.* *J. Am. Water Works Assoc.*, **49**, 881-886 (1957) July.

Corrosion, underground, pipe, cable, cathodic protection, electrical drainage, review, stray currents.—PDA. 15724

### 3.6.8, 3.8.2, 6.3.11, 6.3.2

**Controlled-Potential Reactions of Cadmium and Silver in Alkaline Solutions.** George T. Croft. *J. Electrochem. Soc.*, **106**, No. 4, 278-284 (1959).

A method is described in which overpotential is the independent variable and current density is the dependent variable. The reactions  $Cd + 2OH^- \rightarrow Cd(OH)_2 + 2e$  and  $2Ag + 2OH^- \rightleftharpoons Ag_2O + H_2O + 2e$  were studied. The rate of oxidation of cadmium is a maximum at overpotentials of 18 and approximately 40 mV. The rate of reduction of cadmium and of oxidation of silver are increasing functions of the overpotential.—MA. 18358

## 3.7 Metallurgical Effects

### 3.7.3, 6.3.10, 3.2.2, 3.7.2

**Corrosion-Resistance of Welded Nickel-Molybdenum (Iron) Alloys.** G. N. Flint. *J. Inst. Metals*, **87**, No. 9, 303-310 (1958-59).

Welded nickel-molybdenum-iron alloys of normal commercial purity suffer intergranular corrosion in the zones adjacent to the weld metal when in

contact with many industrial media. This susceptibility arises from the presence of carbides reprecipitated at grain boundaries during cooling after welding. The solubility of carbon in the alloys is so low that it is not practicable to manufacture single-phase alloys of low-carbon content; nor is it possible to form alternative, more stable, carbides. However, additions of vanadium and niobium are beneficial and by combining a vanadium addition with relatively high contents of molybdenum and silicon and maintaining iron at a low level, alloys virtually free from susceptibility to intergranular corrosion in the welded condition can be obtained. (auth)—MA. 18098

### 3.7.3, 3.2.2, 3.7.4, 6.2.5

**Susceptibility of Stainless Steels to Intergranular Corrosion After Heat Treating.** (In Russian.) A. P. Akhshentseva. *Metalloved i Termich. Obrabotka Metallov*, No. 1, 47-49 (1959) Jan. Translation Available from Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, Calif.

Metallographic study of changes in structure of various steels with heat treating conditions and of correlation between structure and intergranular corrosion. Composition ranges studied: 0.06-0.08 percent carbon, 22.3-26.6 chromium, 24-28 nickel, 2-3.5 molybdenum, 0.09-0.61 titanium, 0-3.7 copper. Etchants used; thermal etching technique. Effect of short-time heating between 500 and 1100 C on microstructure and intergranular corrosion. Effect of long-time heating at 650-950 C on phase composition. Cause of susceptibility to intergranular corrosion. 4 figures.—HB. 18251

### 3.7.3, 3.2.2, 6.2.5

**Effect of Prolonged Aging of Austenitic Deposited Metal on Its Tendency to Intercrystalline Corrosion.** (In Russian.) M. A. Gal'perin and V. V. Ardentov. *Avtomat Svarka*, No. 66, 44-51 (1959) Jan. Translation Available from the British Welding Research Assoc., 29 Park Crescent, London, W.I., England.

Resistance of 1Kh18N9T stainless steel plate and of Type 18-8 austenitic-ferritic deposited stainless steels alloyed with molybdenum, vanadium or niobium to intercrystalline corrosion following treatment for 500-1500 hr. at 340-550 C. Change in carbide-phase composition with aging.—RML. 18264

### 3.7.3, 6.2.5, 3.2.2, 4.3.2

**Weld Corrosion in Type 316 and 316L Stainless Steels and Related Problems.** M. E. Carruthers. *Armco Steel Corp. Welding J.*, **38**, No. 6, 259s-267s (1959) June.

Report to WRC High Alloys Committee by Subcommittee on Weld Corrosion summarizes results of survey of corrosion experience in welded Types 316, 316L and 317 in chemical, fabricating and metal-producing industries; and discusses results of accelerated laboratory corrosion tests on Type 316, 316L and 316Cb weld metals. Recommendations are made. Type 316 and 316L sheets and plates gave good service if they were postannealed or stabilized heat treated. When equipment welded with 316 or 316L electrodes was used without benefit of heat treatment, both good and poor performance was experienced depending upon severity of service. Preferential attack at delta-ferrite boundaries in these weld metals was associated with all weld failures studied micro-

scopically. Heat-affected zones in Type 316 sheets and plates failed by intergranular corrosion in several cases. Good corrosion resistance was obtained by repairing pitted Type 316L weld metal with Type 310 coated electrodes. Type 309Cb electrodes also were used to weld Type 316L plates with good corrosion experience "as-welded" in media highly corrosive to Type 316L weld metal. Type 310 Mo weld metal was resistant to corrosion in 65 percent nitric acid but was preferentially attacked when used for repair welding Type 316L exposed to hot 10 percent nitric acid-3 percent hydrofluoric acid solution. Tables, photomicrographs.—INCO. 18323

## 3.8 Miscellaneous Principles

### 3.8.2, 2.3.5, 3.6.5, 3.5.4, 8.4.5

**Chemistry and Electrochemistry of Corrosion Occurring in a Nuclear Reactor.** (In German.) G. H. Cartledge. *Dechema-Monographien*, **30**, No. 411-425, 42-66 (1959).

Theory based on measurements of the electrode potential of platinum and stainless steel in dilute sulfuric acid and at 85 C. Effects of gamma-irradiation and of hydrogen, oxygen or air passed through the electrolyte. 44 references.—RML. 18319

### 3.8.2, 1.6

**Modern Aspects of Electrochemistry No. 2.** J. O'M. Bockris, Editor. Book, 1959, 416 pp. Butterworths Scientific Publications, London.

Contents: H. Falkenhagen and G. Kelbg, "The Present State of the Theory of Electrolytic Solutions"; J. A. Kitchen, "Physical Chemistry of Ion-Exchange Resins"; H. Bloom and J. O'M. Bockris, "Molten Electrolytes"; T. P. Hoar, "The Anodic Behavior of Metals"; M. Green, "Electrochemistry of the Semi-Conductor/Electrolyte Interface."—MA. 18219

### 3.8.2, 3.6.8, 3.6.5

**An Electrochemical Investigation of Hydrogen-Producing Reactions Catalysed by Nickel and Nickel-Palladium Cathodes.** James P. Hoare and Sigmund Schuldiner. *J. Phys. Chem.*, **62**, No. 2, 229-233 (1958).

Hydrogen overvoltage was measured in acid solution, for nickel, palladium and a series of nickel-palladium alloys, and hydrogen-producing mechanisms are discussed. Steady-state, open-circuit potential values for palladium and high-palladium alloys containing dissolved hydrogen were all positive to a Pt/H electrode in the same solution. Nickel-palladium alloys with 32.99 at. percent nickel gave zero potential against the Pt/H reference electrode in the same solution. By use of nickel-palladium-hydrogen ternary alloys it is possible to vary the number of holes/atom in the d-band from ~0 to 0.6. The relative catalytic activity of these metals for the hydrogen-producing reaction is shown to be related to this number, which, in turn is determined by the hydrogen solubility. 23 references.—MA. 18165

### 3.8.2, 6.3.3, 3.6.5

**Activities in the Chromium-Nickel System.** M. B. Panish, R. F. Newton, W. R. Grimes and F. F. Blankenship. *J. Phys. Chem.*, **62**, No. 8, 980-982 (1958).

An electromotive force method for the investigation of activities in the chromium-nickel system is described.

The electrolyte was a molten sodium chloride-rubidium chloride mixture containing ~0.5 percent chromous chloride, and the electrodes chromium and chromium-nickel alloys containing 11-96 at-% chromium. Measurements were carried out at 750 and 965 C. An expected difficulty due to the presence of nickel chloride in the electrolyte from the reaction  $\text{Ni} + \text{CrCl}_3 \rightarrow \text{NiCl}_2 + \text{Cr}$  ( $\Delta F = +23 \text{ k.cal./mole}$  at 1000 K) did not arise because of the long interelectrode-diffusion path. The activity of chromium in (chromium-nickel) alloys (relative to  $\text{Cr}^{\circ}$ ) varies from 0.051 to 0.67 for atom fractions of chromium 0.112-0.378 and the activity coefficient of chromium ranges from ~0.5-1.0 to 30 percent chromium, thereafter rising sharply to the miscibility limit at 40 percent chromium. Oxidation resistance occurs for  $\gamma < 1.0$ , i.e.  $x(\text{Cr}) < 0.3$ . A brief discussion of results in terms of excess entropy of solution is given. Tables, graphs.—MA. 18149

**3.8.3, 6.3.10, 2.3.5**  
A contribution to the Knowledge of the Passivation and Passivity Phenomena of Nickel. Pt. II. Pyridine and Fused Salts. (In English.) R. Piontelli, U. Bertocci and G. Sternheim. Z. Elektrochem., 62, No. 6/7, 772-782 (1858).

Measurements described in Part I are extended to pyridine solution and also to fused nitrates, nitrate-chlorides and chloride baths. The presence of a covering layer does not necessarily involve complete passivity.—MA. 18148

**3.8.3, 6.3.10, 2.3.5**  
Passivity of Nickel. (In German.) K. Schwabe and G. Dietz. Z. Elektrochem., 62, No. 6/7, 751-759 (1958).

An apparatus is described for measuring the potential of a nickel electrode which can be polished with a corundum disc in situ. Direct dependence on  $\text{H}^+$  activity is shown. The results are explained as mixed anodic and cathodic processes. The polished electrode shows passivity in sulfate solution and in solution of perchlorates containing sulfuric acid; a chemisorbed oxygen layer forms. The influence of anions on the passivity is discussed. 16 references.—MA. 18160

**3.8.3**  
**Corrosion and Passivation.** (In French.) U. F. Franck. Corrosion et Anticorrosion, 7, 83-97 (1959) March.

Passive state results from very thin films which are formed on the metal surface. The reaction of passivated metals toward corrosion is primarily determined by chemical and electric properties of the film and not by the properties of metal itself. 62 references.—RML. 18272

**3.8.3, 6.2.2**  
**Adsorption Theory of Passivity and the Flade Potential.** Herbert H. Uhlig. Z. Elektrochem., 61, No. 6/7, 626-632 (1958).

From quantitative data reported by various investigations, it is concluded that the primary passive film on iron has the following properties: (1) 25-100 Å thick, (2) equivalent to 0.01 coulomb/cm<sup>2</sup>, (3) can oxidize  $\text{CrO}_4^{2-}$  to  $\text{CrO}_4^{-}$ , (4) follows the (Flade) potential relation  $-0.58 + .058 \text{ pH}$  (volts). All these properties cannot be accounted for by any of the known oxides of iron. In better accord with the facts is a proposed passive film composed of chemisorbed atomic and molecular oxygen

supplemented perhaps by  $\text{OH}^-$  and water.—BTR. 18200

### 3.8.3, 6.3.10, 2.3.5

A Contribution to the Knowledge of the Passivation and Passivity of Nickel. Pt. I. Aqueous Solutions. (In English.) R. Piontelli and G. Serravalle. Z. Elektrochem., 62, No. 6/7, 759-772 (1958).

Passivation of nickel was studied by electrode voltage-measurements, against standard references electrodes, with rectangular current pulses. Temperature, pH, and current density were varied, and fluoride, sulfate, sulfamate and perchlorate solutions used. At ordinary temperature the anodic behavior of nickel varies with pH, temperature and current density, as does its electrochemical inertia. Possible causes of passivity are discussed. Distinction between local and average behavior is stressed. 19 references.—MA. 18147

### 3.8.3, 6.2.2

Anodic Passivation of Iron. E. S. Snavely, Jr. and N. Hackerman. University of Texas. Paper before Symposium on Charge Transfer Processes, Univ. of Toronto, Sept. 4-5, 1958. Can. J. Chem., 37, No. 1, 268-275 (1959) Jan.

Study of anodic passivation of iron in solutions of 0.1M sodium sulfate at pH of 3.0. Mass balance of iron equivalent to coulombs required for passivation and iron found in solution after passivation showed that 0.003 coulomb/cm<sup>2</sup> is involved in passive layer. Flade arrest does not appear in decay traces of anodic potential unless potential of at least 1.2 V (E<sub>H</sub>) is achieved on charging electrode, indicating incomplete coverage by passive layer below 1.2 V. Partial cathodic reduction followed by anodic reformation of passive layer at Flade arrest showed that 1st charging current involved in reformation is highly efficient even when as much as 70% of Flade arrest is reduced. Only fraction of passive layer is represented by coulombs involved in Flade arrest and after partial reduction remainder of passive layer disintegrates spontaneously. Addition of chelating agents to electrolyte has adverse effect on passivation of ferrous ion. Graphs.—INCO. 17702

### 3.8.3, 6.3.3

Passivity of Chromium in Dilute Sulfuric Acid. (In German.) Th. Heumann and F. W. Diekötter. Z. Elektrochem., 62, No. 6/7, 745-750 (1958).

Potential/time curves were measured for electrolytic chromium by a galvanostatic method. The electrolyte was dilute sulfuric acid with 0.1 M-potassium sulfate at pH 0.6-2.2 and 20 C. Potential increases initially, shows a retardation at 250 mV, and reaches the final value with decreasing slope. The relation  $i_{io} = K/t$  is observed, where  $i_{io}$  = limiting current decreasing with pH,  $K$  = constant = 0.0023 amp. sec./cm<sup>2</sup>,  $t$  = passivation time. Passivating current density is greatly influenced by hydrogen evolution. Results are compared with those for iron and iron-chromium alloys. 10 references.—MA. 18009

### 3.8.4, 3.5.5

Formation of Porous Oxides on Metals. D. W. Aylmore, S. J. Gregg, and W. B. Jepson. University of Exeter. J. Electrochem. Soc., 106, 1010-1013 (1959) Dec.

Porosity of the oxide scales formed on a number of metals during oxidation in dry oxygen has been investigated by measurements of specific surface and of density. The oxides formed during the

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linear oxidation of the eight metals: calcium, cerium, lead, magnesium, niobium, thorium, tungsten, and uranium are porous whereas those formed during the parabolic oxidation of copper and cobalt are impervious to oxygen gas.

18243

**3.8.4, 6.2.5**

**Mechanism of Oxidation at High Temperatures of Austenite Steel with 18 percent Cr-8 percent Ni.** (In French.) Jacques Bénard, Jean Hertz, Yves Jeannin, and Jean Moreau. Compt. rend., **248**, 2095-2097 (1959) Apr. 6.

Oxidation of chromium-nickel steel was investigated at 1050°C by a kinetic study and microscopic, x-ray, and electron diffraction examination. The results show that the oxidation has an initial period of slow oxidation. This period of oxidation follows a parabolic law. The oxide film formed appears to protect the alloy for a time. Then the second period of oxidation occurs which is characterized by a rapid oxidation. This period continues until the elimination of iron from the chromium-poor layer is achieved. Then the third period, characterized by a slow oxidation, appears.—NSA.

18261

**3.8.4, 3.2.3**

**Transitions from Parabolic to Linear Kinetics in Scaling of Metals.** E. W. Haycock. Shell Development Co. J. Electrochem. Soc., **106**, 771-775 (1959) Sept.

A general rate equation is derived for a model of a barrier scale layer being formed by a diffusion process and simultaneously being depleted at a constant rate by a secondary process. The

equation, derived to satisfy rate curves for sulfide scaling of iron in hydrogen sulfide, is shown to be applicable to the oxidation of aluminum and hafnium over ranges of conditions where two or three rate equations have been used previously to explain the experimental data. The validity of this equation emphasizes the importance of secondary processes, such as scale recrystallization, in determining the over-all rate of high-temperature corrosion reactions.

**3.8.4, 6.3.13, 2.3.5**

**Kinetics of Aqueous Oxidation of Tantalum from Chronopotentiometric Measurements.** George B. Adams, Jr. and Eugene Y. K. Mak. Oregon Univ. U. S. Atomic Energy Commission Pubn., AECU-3977, 1958, 19 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington, D. C.

Theoretical mechanisms and growth laws for the initial oxidation of metals are briefly reviewed. The oxidation of tantalum was studied chronopotentiometrically at temperatures between 0 and 40°C in an oxygen-free electrolyte. The probable rate determining steps for the two different growth laws observed are deduced by comparison of the time dependence of the experimentally determined local oxidation current to theoretical local current-time functions derived from assumed growth laws. Tantalum oxidizes initially according to a direct logarithmic law, the slow step being tunneling of electrons through the oxide film. At 40°C a transition from a direct logarithmic to a parabolic growth law is observed. The interpretation is that the diffusion of electrons which have been injected thermionically into the oxide film becomes rate determining

at film thicknesses for which current due to electron tunneling becomes negligible. (auth).—NSA.

18067

**3.8.4, 3.4.6, 3.8.3, 6.6.9**

**Passivity During the Oxidation of Silicon at Elevated Temperatures.** Carl Wagner. J. Applied Physics, **29**, No. 9, 1295-1297 (1958).

Wagner reviews theoretically the dependence of the rate of oxidation of silicon in oxygen-helium mixtures on gas composition at its melting point. At low oxygen contents, no solid silicon dioxide is formed and the rate of attack  $\propto$  the oxygen partial pressure in the gas mixture. Above a critical oxygen pressure, the formation of solid silicon dioxide produces passivity and the rate of attack, under steady-state conditions may drop by a factor of  $10^n$ .—MA. 18154

**5. PREVENTIVE MEASURES****5.4 Non-Metallic Coatings and Paints****5.4.5**

**Silicone Protective Coatings for the Steel Industry.** R. C. Hedlund. Iron Steel Eng., **34**, 109-112 (1957).

Use of silicone and silicone/synthetic resin paint and varnish coatings is reviewed. Their water repellency was referred to in the discussion.—RPI. 17870

**5.4.5, 6.4.2**

**Organic Finishing of Aluminium and its Alloys.** L. F. Spencer. Metal Finishing, **56**, No. 8, 58-61, 69 (1958) August.

In the organic finishing of aluminum alloys, preparation of surface can be considered almost as important as the selection of the correct organic coating. The different methods of surface preparation, including the use of wash primers, are described. A zinc chrome primer formulated with phenolic and/or alkyd resin base is satisfactory for severe service. Vinyl-base primers pigmented with zinc chrome are also recommended. Lead-containing pigments should be avoided. For final finish coats, synthetic resin formulations of the laquer or enamel types are usually employed. Bituminous paints are also commonly used for protection in corrosive environments.—RPI. 17832

**5.4.5, 8.1.2**

**Paint is Preserving Bridges, but There are Still Failure Points.** F. D. Johnson. Can. Paint & Varnish, **31**, No. 6, 30-31, 46, 51, 58, 70, 72 (1957).

Role of paint in protecting Brooklyn Bridge and the Eiffel Tower is described. On the bridge the priming coat is based on a red lead/linsen oil composition; white lead is the major pigment in the finish coat. Repainting of the bridge takes place once every 8-10 years, while the Eiffel Tower is done about once every seven years. The Eiffel Tower authorities have set up a kind of dictionary of failure terms used in their contracts and these terms are quoted.—RPI. 17860

**5.4.5, 6.2.4**

**Protecting Sheet-Steel Products.** A. N. Laubscher and C. P. Larrabee. Paint Var. Prod., **47**, No. 9, 35-37, 89 (1957).

A protective coating such as vinyl on sheet steel prior to fabrication reduces deterioration. The surface life of a paint film depends upon the corrosion resist-

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ance of steel to which the paint is applied. The service life of paint film is greater when application is made on "clean" steel than when it is made on hot rolled, weathered, wire-brushed steel. Under marine atmospheric conditions, a paint system having a zinc-chrome primer gives longer service life on a high-strength low-alloy steel than does a paint system having a red lead/linsed oil primer.—RPI. 17924

**5.4.5, 6.4.4**  
**Magnesium Finishing.** Dow Chemical Company. Industrial Finishing (Brit.), 9, Nos. 111, 112, 792-796, 801; 865-866, 868-870 (1957).

Various methods of surface treatment of magnesium are described. Primers for magnesium should provide good adhesion to the metals and are commonly pigmented with zinc chrome as corrosion inhibitor. Some wash primers are being used successfully on magnesium, but they should be employed with caution. To prevent galvanic attack between magnesium and dissimilar metals, paint films should be applied to both the dissimilar metal and magnesium and should be alkali-resistant.—RPI. 17859

**5.4.7, 1.3**  
**Flame Spraying of Non-Metallic Protective Coatings on Metal Surfaces.** (In German.) H. Reiningher. Metalloberfläche, 11, No. 12, 393-400 (1957).

A review, dealing with: pretreatment of the metal surface; spraying pistols and ancillary equipment; materials (plastics) for spraying and methods of producing the necessary powders; properties and application of the coatings. 72 references.—MA. 17988

## 5.8 Inhibitors and Passivators

**5.8.4, 5.7.1, 4.6.1**  
**Part Played by Corrosion Inhibitors in the Treatment of Water.** (In French.) E. L. Streathfield. Metaux Corrosion-Industries, 33, 420-430 (1958) October. Survey of the cold and hot softening and deionizing processes, effects on carbonic anhydride and oxygen. Mechanism and distribution of corrosion. The use of anodic, cathodic or organic inhibitors, especially sodium benzoate and amines.—BTR. 17830

**5.8.4, 4.3.2, 2.3.4**  
**New Organic Inhibitors for Protection of Steel from Acidic Corrosion.** (In Rumanian.) I. V. Nikolesku, G. Landau and A. Sachevyanu. Rev. Chim., Acad. Rep. Populaire Roumaine, 2, No. 1, 49-59 (1957).

Inhibiting action of high molecular weight compounds with a large number of functional groups on the corrosion of sheet steel at 20°C and 40°C was studied by a gravimetric method with totally submerged test pieces. 17886

**5.9.2, 4.3.2, 4.3.3, 6.4.2**  
**Chemical Polishing of Aluminum and Its Alloys.** Emanuel G. Popescu. Ind. Usoara (Bucharest), 5, 145-148 (1958); Chem. Absts., 53, No. 2, (1959) Jan. 25. Polishing baths containing either phosphoric acid or ammonium bifluoride-nitric acid mixtures give very good results, but strongly alkaline solutions are too hard to handle. In order to avoid excessive corrosion, stainless steel containers are recommended for solutions containing phosphoric acid whereas polyethylene-coated ordinary steel vats are required for the bifluoride base

solution. Chemical polishing is superior to other polishing methods.—ALL. 17566

### 5.8.4, 3.4.2

**Studies on Amine-Type Corrosion Inhibitors.** Rept. No. 14. Relationship Between Corrosion-Inhibitors and Catalyst Poison. (In Japanese.) Seiichi Fujii and Kenzo Kobayashi. Corrosion Engineering (Japan), 8, No. 2, 3-6 (1959) Feb.

If a dilute solution of a corrosion-inhibitor is allowed to remain in contact with a limited amount of catalyst, such as nickel hydrogenation catalyst, the catalyst will be adsorbed on the surface by the inhibitor. The catalyst loses its activity by being shielded with the inhibitor film.

The partial catalytic hydrogenation of

croton-aldehyde in an autoclave proceeds as follows: (1)  $\text{CH}_2\text{CH} = \text{CHCHO} \xrightarrow{\text{H}_2} \text{CH}_3\text{CH}_2\text{CH}_2\text{CHO}$   
(2)  $\text{CH}_2\text{CH} = \text{CHCHO} \xrightarrow{\text{H}_2} \text{CH}_3\text{CH} = \text{CHCH}_2\text{OH}$

The former reaction is taking place mainly at lower temperature and the latter at higher temperature. With non-shielded nickel catalyst the reaction proceeds at about 40°C and the partially hydrogenated product is almost butyraldehyde which identifies that the reaction proceeds only as shown in the equation (1). With  $\text{C}_{16}\text{H}_{33}\text{NH}_2$ -treated catalyst the reaction proceeds at about 80°C and the product contains crotyl-alcohol which identifies the equation (2). With  $\text{C}_{15}\text{H}_{31}\text{CONH}_2$ -treated catalyst the reaction proceeds at 130°C and crotylalcohol is predominant.

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So, the inhibitor film chemi-sorbed onto the metal surface will leave from there at definite temperature, as given by type of functional group. The treated catalyst revives at the critical temperature of the desorption and the catalytic reaction can proceed as contact with non-treated catalyst. 17843

#### 5.8.4

**Studies on Amine-Type Corrosion Inhibitors.** Rept. No. 15. Inhibitors with Branched Hydrocarbon Radicals. (In Japanese.) Seiichi Fujii and Kunitsugu Aramaki. *Corrosion Engineering*, **8**, No. 4, 1-4 (1959) April.

High molecular-weight organic corrosion inhibitors are consisting of two functional atomic groups. The one functions as adsorbent, another is hydrocarbon radicals which resist the corrosive matters. Metals are protected from the penetration of corrosive materials by these barriers which are built up with the hydrocarbon radicals.

With inhibitors of high molecular-weight, a densely packed film with the hydrocarbon chains oriented outward, is formed on the metal surface. But with those inhibitors whose hydrocarbon chains are branched at nearer position to adsorbent, worse corrosion inhibition is gained than with not branched ones.

For these reasons, we concluded that decreasing inhibition were indebted (1) to inter-molecular interference with regular orientation of inhibitor molecules on the metal surface and (2) to inner molecular interference with adsorbent to adsorb onto the metal surface. 17868

#### 5.8.4, 6.2.2, 3.4.9

**Nitroamines in the Protection of Iron Against Corrosion.** (In Spanish.) Mario Petit and Fernando Estalella. *Rev. Cien. Apli. (Madrid)*, **12**, 293-298 (1958) July-August.

Ferrous metals wrapped in paper impregnated with nitrite of dicyclohexylamine or tri-ethanolamine in amounts in excess of 0.05 g per sq dm effectively protected against oxidation produced by atmosphere saturated with water vapor at a temperature of  $22 \pm 2$  C. Procedures for preparing these corrosion inhibitors and their properties. 20 references.—MR. 17544

### 5.9 Surface Treatment

#### 5.9.1

**General Practices of Metal Surface Prepaint Preparation.** J. H. Geyer. *Official Digest*, **29**, No. 389, 533-541 (1957).

A critical review is made of sandblasting, abrading with steel wool, washing with alkali, vapour and solvent degreasing, phosphating and anodizing, as painting pretreatments for metals.—RPI. 15846

#### 5.9.2

**Effect of Halide Addition to the Sulfuric acid Pickling Bath.** M. Haftel and W. L. Reichel. Industrial Test Laboratory, Philadelphia Naval Shipyard, U. S. Navy, February, 1957, 27 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 131794).

Sodium halide additions to the standard sulfuric acid pickling bath reduces acid attack on base metal, especially for special treatment steel, according to this report. The effect is described as more pronounced with increases in pickling time. The additions, however, had no apparent effects on scale removal rate,

surface appearance, or surface passivation and paint adhesion. Halide concentrations of 0.1 percent are recommended. Because there is little difference in inhibiting properties of halides, the low-cost sodium chloride is considered most suitable.—OTS. 17852

Finishing, **22**, No. 9, 32-37 (1958).

Chromate coatings on zinc may be used as final finishes to protect against corrosion and impart color, or as pre-paint treatments to prevent reaction between metal and paint, to provide a good base for paint and to prevent the spread of under-film corrosion. Chromate coatings are usually deposited from acidic aqueous solutions and the condition of the zinc surface has a marked influence on the condition of the coating deposited. Other factors influencing deposition include anion/cation balance in the chromate solution, time of metal immersion, temperature and pH of the solution, agitation of the bath, time between dipping and rinsing of the metal and adequacy of the rinsing procedure.—RPI. 17723

#### 5.9.4, 2.3.2

**Deterioration of Anodized Aluminum Exposed to Corrosive Environments.** J. M. Kape. *Electroplating & Metal Finishing*, **12**, No. 2, 41-46, 59 (1959) Feb.

Reviews literature on resistance of anodized aluminum to surface deterioration and discusses best methods available of producing resistant anodic film. Methods of testing surface deterioration (weathering and chemical action) are considered from theoretical and practical standpoints. Saline tests using sodium chloride or sea water alone, and saline tests modified with acetic acid, hydrogen peroxide or sulfur dioxide are evaluated. Tests which are considered to indicate degree to which properly-sealed coating would perform under service conditions are: C.R.L. beaker test, Kesternich test, and Aluminium Labs. test. Resistance of anodized coatings to various chemicals is reviewed and tabulated data presented. Some agreement between salt spray tests, salt-acetic acid tests, various sulfur dioxide tests and outdoor service exposure has been achieved. In contrast to electrodeposits, however, anodic film is corrosion tested by measurement of efficiency of sealing, and measurement of overall protective power of anodic film relative to base metal.—INCO. 17711

#### 5.9.2, 2.3.9

**Chromic Acid Rinse Treatment.** S. L. Eisler. *Ind. Finishing (Brit.)*, **9**, No. 111, 818-821 (1957).

A radiometric technique for measuring the action of chromic acid in rinse solutions on metal surfaces is described. From the quantitative measurements obtained the following conclusions were reached: chromic acid adsorption varies as the solution concentration; solutions containing phosphoric acid have increased rates of adsorption over solutions of equal chromic acid concentration but without added phosphoric acid the amount of desorption of chromic acid is small compared with the total adsorbed.—RPI. 17877

#### 5.9.2, 5.9.4, 2.3.9, 4.3.3

**New Stripping Solution for Phosphate Coatings.** S. L. Eisler. *Ind. Finishing (Brit.)*, **9**, No. 112, 878, 880, 883, 885-887 (1957).

Details of tests conducted using radiometric techniques to determine the effectiveness of a stripping solution containing sodium hydroxide and sodium cyanide alone and with tetra-sodium ethylenediamine-tetra-acetate as a separating agent are given. A solution containing 24 oz/gal of sodium hydroxide and 12 oz/gal of sodium cyanide with an additional 12 oz/gal of the separating agent is recommended for the removal of zinc, iron and manganese phosphate coatings. The rephosphating efficiencies were found to be about 100 percent and the corrosion resistance of the rephosphated material was found to be equal to that of the material phosphated for the first time.—RPI. 17851

#### 5.9.4, 6.3.19

**How to Produce High-Quality Zinc Chromate Coatings.** J. Stella. Products

described discussed is referred

6.3.15, 4.3. Rate of titanium in sition of Strauman chem., 62

Titanium from a p face of porcelain. Weight-temperatur breaks of soon as TiO<sub>2</sub>. Ti oxidize fresh par deposit a 44 cm<sup>2</sup> at 800 The case mole. 44 1

6.3.17, 3.5. Problem ZPR-6, R. Lab. U. S. Pubn., A 1956 (Dec Available Services, Washington

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6.3.17, 4.6. Corrosion nium)-Col Nelson, V Hopkins, Energy C 19, 1945 (pp. Available Services, Washington

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17594

Tests were made of the corrosion resistance of titanium in solutions of sulfuric acid with and without anode protection.—NSA. 17594

**6.3.15, 8.8.1 Chemical Plant in Titanium.** R. J. Watkins. *Industrial Chemist*, **34**, No. 400, 282-286 (1958).

High corrosion resistance of titanium makes it a suitable material in the construction of chemical plant. Owing to its high cost, titanium linings are used in large plant equipment. Mechanical and physical properties of titanium and titanium-aluminum alloys containing manganese, tin or vanadium are

13.17, 2.3.4 Aqueous C. J. B. Schi Electro 49 (1959) The me

## CORROSION ABSTRACTS

described. Future uses of titanium are discussed and work on titanium anodes is referred to.—MA. 17927

## 6.3.15, 4.7, 3.8.4, 3.4.3

**Rate of Oxidation (Corrosion) of Titanium in Molten Salt and the Composition of the Oxidation Products.** M. E. Straumanis and Ch. Chiou. *Z. Elektrochem.*, **62**, No. 2, 201-209 (1958).

Titanium wire spirals were suspended from a platinum wire beneath the surface of molten sodium chloride in a porcelain crucible open to the air.

Weight-loss/time and weight-loss/temperature curves were linear. The oxygen-enriched surface of the titanium breaks off and disperses in the salt as soon as its composition approaches  $TiO_{2.5}$ .

The dispersed particles slowly oxidize to titanium dioxide-rutile. As fresh particles touch the crucible they deposit a titanium layer.

Weight losses were 0.00607, 0.0104 and 0.0136 g/hr/cm<sup>2</sup> at 800, 950 and 1000 C, respectively.

The ease of oxidation is explained by the low activation-energy of 19,140 cal/mole. 44 references.—MA. 17845

6.3.17, 3.5.9, 3.4.6, 8.4.5

**Problem of Oxidation of Uranium in ZPR-6.** R. C. Vogel. Argonne National Lab., U. S. Atomic Energy Commission Pubn., ANL-RCV-SL-1120, Nov. 7, 1956 (Declassified March 9, 1957), 2 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C.

The effects of temperature on uranium oxidation rates in pure oxygen are shown.—NSA. 18006

6.3.17, 4.6.5, 3.7.2, 3.5.9

**Corrosion Testing of Tuballoy (Uranium)-Columbium Alloys.** Frederick Nelson, W. W. Binger and Joyce M. Hopkins. Chicago Univ. U. S. Atomic Energy Comm. Pubn., CT-3052, June 19, 1945 (Declassified Feb. 18, 1957) 36 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C.

Hot rolled and variously heat treated uranium-niobium alloys (2, 4 and 6 wt. percent) were corrosion tested in relatively pure, air-free water. The corrosion resistance of similarly heat treated alloys in distilled water at 178 C increased with niobium content. Heat treatment also had an important effect on corrosion resistance. The effects of niobium content and of heat treatment on the corrosion resistance of the alloys in boiling, distilled water at 100 C were similar to those at 178 C. It is estimated that pimple growth on a leaky, unbonded, canned slug of the most resistant alloy, tested isothermally in water at 100 C, would be about 5 mils in 90 days. Air free, 1 percent sodium chloride solution attacked the water quenched 6 wt. percent niobium alloy somewhat more rapidly at 178 C than did distilled water. This alloy was very severely attacked by air-free 1 percent sodium fluoride solution at the same temperature. Aerated water containing mixed electrolytes and a very small concentration of hydrogen peroxide attacked the water quenched alloys somewhat faster at 50 C than boiling water did at 100 C. (auth)—NSA. 17925

6.3.17, 2.3.4, 3.8.4

**Aqueous Uranium Corrosion at 100 C.** J. B. Schroeder, D. A. Vaughan and C. M. Schwartz. Battelle Memorial Inst. J. Electrochem. Soc., **106**, No. 6, 486-489 (1959) June.

The mechanism of aqueous uranium

corrosion has been studied using weight loss and hydrogen evolution measurements. Microscopic x-ray diffraction and chemical analyses were made of the corrosion product. The results indicate that uranium corrodes by forming an oxide of the type  $UO_{2+x}$ . The lack of chemical balance reported by earlier workers is explained by the existence of metallic uranium in the oxide layer. 17800

## 6.3.17, 4.6.4

**Evaluation of Grinding Coolants: Corrosive Effects on Uranium Metal.** Fred H. Meyer, National Lead Co. of Ohio. U. S. Atomic Energy Commission Pubn., FMPC-192, Feb. 24, 1953 (Declassified April 3, 1957) 11 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C.

A series of grinding coolants were tested for their corrosion effects on uranium metal. Both undiluted and various concentrations of coolant-in-water mixtures were used. A standardized test arrangement and rating system were used for evaluating and comparing the coolants. The results are tabulated. (auth)—NSA. 17926

## 6.3.17, 3.8.4, 2.3.9

**Oxidation of Uranium in Air: A Review of Progress to February 27, 1953.** K. Q. Bagley and D. S. Oliver. Culcheth Labs. U. S. Atomic Energy Commission Pubn., RDB(C)/TN-32, June 5, 1953 (Declassified Sept. 3, 1957), 42 pp. Available from Culcheth Labs., Culcheth, Lancs, England.

The kinetics of the oxidation of uranium in air have been investigated in the temperature range 150 to 500 C by gravimetric, metallographic and x-ray methods. Data were obtained on the rate of oxidation, the composition of the scale, and the superficial changes in the scale at different temperatures. A brief survey was made of the rate of reaction of uranium in pure nitrogen. The data indicate that the mode, as well as the degree of oxidation, changes as the temperature rises and activation energies have been evaluated for the processes successively controlling the rate of reaction. (auth)—NSA. 17871

## 6.3.19, 3.6.8, 3.8.3, 3.6.5, 4.3.3

**Electrochemical Behaviour of Zinc in Alkaline Solutions. Pt. I. Constant Current Measurements.** Indra Sanghi and W. F. K. Wynne-Jones. Proc. Indian Acad. Sci., A, **47**, No. 2, 49-64 (1958).

The cathodic and anodic polarization of zinc in different concentrations of alkaline solutions under constant-current conditions were studied. The changes of zinc electrode-potential with reference to a  $Hg|HgO|KOH$  system were measured in KOH and zincate solution. Time/potential curves indicate that anodic zinc passivates at a certain stage showing a sudden rise in potential by as much as 3.4 V. Before passivation the main anode reaction is the formation of  $Zn^{2+}$ ; after passivation gas evolution occurs. No definite relationship between current density and passivation time was found; several reasons including migration and non-linear diffusion effects, are suggested and discussed. Zinc differs in behavior in concentrated alkaline solution (i.e. 0.5-5N) and in diluted solution (0.2-0.01N). In dilute solution the open-circuit rest-potential of zinc is indefinite and may vary by as much as 500-600 mV, while the maximum passivation-jump in potential is approximately 2 V only. The passivation and

decay of current on breaking the circuit are slower than in the case of concentrated solution. Color changes of the zinc electrode during polarization are described and discussed. 30 references.—MA. 17689

## 6.4 Non-Ferrous Metals and Alloys—Light

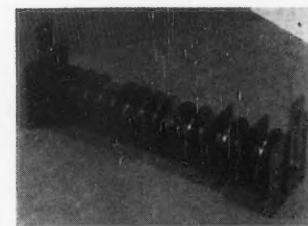
## 6.4.2, 3.2.2, 3.6.8

**Intercrystalline Corrosion of Copper-Containing Aluminum Alloys.** (In Russian.) S. E. Pavlov and S. M. Ambartsumyan. *Korroziya i Zashchita Metal. Sbornik (U.S.S.R.)*, **1957**, 199-217; Chem. Absts., **52**, No. 22, 19855 (1958) Nov. 25.

Corrosion of polished surfaces of cast aluminum alloys containing 4 percent and 12 percent copper, and of duralumin, immersed in 0.01 percent and 3 percent sodium chloride solutions, was observed under a microscope. Intercrystalline corrosion (I) was observed only on surfaces on which continuous chains of  $CuAl_2$  precipitation were present. However, in such cases the surface could be subdivided into two types of areas: on one type, localized corrosion with formation of many dark spots began, gradually spread, and covered the surface with a dark, protective film; on the other type areas, usually circular and light, there were no small dark spots, but hydrogen evolution was observed in the center of the area. At a later stage, I became apparent, but was confined to the light areas. Coupling with pure aluminum eliminated light areas, while additional coupling to pure copper caused an increase in the light areas and an intensification of I. It is concluded that I occurs only under con-

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ditions of hydrogen depolarization, which probably begins in poorly aerated micropits in the surface, while oxygen depolarization produces protective films and does induce I. It is proposed that the micropits are generated by shrinkage in casting. The hydrogen depolarization is triggered by negative difference effects caused by local corrosion microelements.—ALL. 17993

#### 6.4.2, 4.3.3, 4.4.8

**Investigations on the Corrosion Behavior of Aluminum and Aluminium Alloys Towards Organic and Inorganic Bases.** (In German.) A. Bukowiecki. Werkstoffe und Korrosion, **10**, No. 2, 91-105, (1959) February.

A comparative study of the corrosive effects of organic and inorganic bases on aluminum materials is presented. Corrosion tests were carried out at 20°C temperature with exposures as long as 73 hours, whereby it has been established that the aggressivity of the bases investigated decreases in the following order: sodium hydroxide, methylamine, ethylamine, n-butylamine, ammonia, triethanolamine, sodium carbonate, calcium hydroxide and hydrazine. Among the aluminum materials tested 99.3 percent aluminum exhibited the highest sensitivity to corrosion followed in a decreasing order by Raffinal (>99.9 percent), Anticorodal (soft), Avional 23 (aged), Perunal (aged), and Peraluman 30 (soft). For every mixture of a base and water, there is a critical concentration at which the intensity of the attack reaches its maximum. Bases of high concentration cause but little corrosion and anhydrous bases are rather inactive. There is no distinct relationship between aggressivity of the bases and either their pH values, or their electric conductivity.—ALL. 17826

#### 6.4.2, 3.4.8

**Corrosion Resistance of Aluminum Materials to Aqueous Hydrogen Sulfide Solutions.** (In Czech.) Karel Smrk, Ivan Sekera and Vladimir Seifert. Chem. prumysl (Czechoslovakia), **8**, 297-301 (1958). Chem. Absts., **52**, No. 22, 19846 (1958) Nov. 25.

In water solution in equilibrium with hydrogen sulfide at one atmosphere, 99.5 percent aluminum and aluminum alloys with magnesium, manganese and silicon corrode very little, the rate decreasing with rising temperature. The 20°C values (mm/yr) were  $1.1 \times 10^{-5}$ ,  $5.6 \times 10^{-5}$ ,  $9.7 \times 10^{-5}$  and  $2.5 \times 10^{-5}$ , respectively. At hydrogen sulfide pressure of eight atmosphere, corrosion rates at 20°C were unchanged, but were higher at 100°C. With partial sample immersion at 100°C, deposition of elemental sulfur at the solution surface led to much more severe attack. Aluminum welds showed no tendency to corrode in hydrogen sulfide solution.—ALL. 17738

#### 6.4.2, 3.2.2, 3.7.3, 5.4.5

**Intercrystalline Corrosion and Stress Corrosion of Tubes of Alloy D16-T (Duralumin).** (In Russian.) S. E. Pavlov and A. F. Maslova. Korroziya i Zashchita Metal., Sbornik (U.S.S.R.), **1957**, 218-235; Chem. Absts., **52**, No. 22, 19855-19856 (1958) Nov. 25.

Tubes of alloy D16-T (duralumin) had regions which were susceptible to intercrystalline corrosion (I) in 3 percent sodium-Cl plus 1 percent hydrochloric acid solution, even if the time interval between heat-treating and quenching, under shop conditions, was

only three sec. Quenching in a nitrate bath was especially conductive to I. Tubes which were not susceptible at stress equal to 90 percent of the yield stress. Plastic deformation in calibration after the heat-treated induced sensitivity to stress corrosion cracking. Anodized film promoted I, by concentration of damage in the cracks in the film. Hence, to protect against I, anodized films must be further reinforced by organic coatings.—ALL. 17991

#### 6.4.3, 8.8.5

**Fabrication and Properties of Commercially Pure Beryllium.** J. Williams. Metallurgical Reviews, **3**, No. 9, 1-44 (1948).

Refining of beryllium by thermal reduction and by fused-salt electrolysis is reviewed. Section on fabrication covers melting and casting, production of beryllium powder, cold compacting and sintering, hot-compacting processes, extrusion, rolling, forging, coining and swaging, machining, and joining. Deformation of beryllium single crystals is considered. Room temperature mechanical properties of polycrystalline beryllium reviewed are tensile strength and elongation, tensile modulus, Poisson's ratio, impact and notched-tensile properties, torsion and shear, and compression. Elevated temperature properties considered include short-time tensile properties, stress-rupture, creep, compression, torsion and impact properties. Heat treatment of beryllium is dealt with. Section on corrosion covers behavior of beryllium in gases, in water and in liquid metals. Problem of brittleness of beryllium is reviewed. Tables, graphs, 70 references.—INCO. 17721

#### 6.4.4, 3.4.6

**Behavior of Magnesium and Two of Its Alloys Heated in Carbon Dioxide.** (In French.) M. L. Bousson, R. Darras and D. Leclercq. Rev. mét., **56**, No. 1, 61-67 (1959) Jan.

Experiments described consisted in heating magnesium and two alloys, magnesium-0.7 chromium and magnesium-0.7 percent zirconium-0.9 percent zinc, together with carbon dioxide in an autoclave. Duration of the tests was 3000 hours, the pressure 15 kg/sq cm and the temperature varied between 400 and 500°C. For all three materials, weight increase below 450°C was very slight. However, at higher temperatures the magnesium-zirconium-zinc alloy is subject to rapid sublimation of its zinc content. It was noticed that while the effect of gas pressure on the corrosion process was quite substantial, the influence of small oxygen, nitrogen and air contents present in the carbon dioxide was negligible.—ALL. 17787

#### 6.4.4, 4.3.5, 3.4.9, 3.8.4, 3.5.9

**High Temperature Oxidation of Magnesium in Dry and in Moist Oxygen.** S. J. Gregg and W. B. Jepson. J. Inst. Metals, **87**, No. 6, 187-203 (1959) Feb.

With the aid of the thermal balance the oxidation of magnesium sheet has been investigated between 350 and 600°C in dry oxygen and in moist oxygen at four different humidities. Five different modes of preparation of the surface of the metal have been used. The oxidation curves of weight-gain against time are characterized by an induction period (during which lateral growth of white oxide across the specimen is observed) followed by a linear branch. In dry oxygen at 325°C and above, a second

linear branch is found. Presence of moisture lowers the temperature at which the oxide film ceases to be "protective" shortens the length of the induction period, increases the rate of linear oxidation and inhibits the development of the second linear branch. The experimental energy of activation decreases with increasing humidity of the oxygen. In dry, but not in moist oxygen the course of the oxidation is very sensitive to the method of surface preparation. The reaction mechanism is discussed and a model for linear oxidation (based on a stationary state in which the rate of thickening and of cracking of the oxide film are equal) is suggested. The effect of moisture in promoting oxidation is ascribed to the incorporation of hydroxyl ions in the oxide film.—ALL. 17751

## 6.6 Non-Metallic Materials

#### 6.6.6, 3.5.9

**Ceramic Materials for High-Temperature Applications in the Chemical Process Industries.** M. Jack Snyder. Battelle Memorial Inst., Columbus, Ohio. Chem. Eng. Progress, **54**, No. 11, 65-6 (1958) Nov.

Recently developed high-temperature processes (above 1500°C) which require ceramic containment and the ceramic materials used are discussed. Tables showing upper operating temperatures of commercial ceramics and pure compounds and their uses and limitations are given.—NSA. 17744

#### 6.6.6, 4.3.3

**Initial Report on the Corrosion of Fused Titania and Synthetic Sapphires in Uranyl Sulfate Solutions at 250°C.** J. L. English. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn. CF-52-5-33, May 5, 1952 (Declassified Feb. 14, 1957), 5 pp. Available from Office of Technical Services, Washington, D. C.

Samples of fused titania and synthetic sapphires submitted for corrosion behavior evaluation in uranyl sulfate solutions at 250°C showed weight losses of 0.2 to 0.3 mil/year for titania and 0.7 to 0.8 mil/year for sapphires. Of the numerous ceramics and related materials studied thus far in uranyl sulfate solutions, these have offered the most promising corrosion behavior.—NSA. 15232

#### 6.6.8

**Plastic Panel Coverings: Handle Tough Plant Conditions.** P. N. Cherenisoff. Alsynite Co. of America. Chem. Eng., **65**, No. 9, 152, 154, 156 (1958) May 5.

Translucent fiberglass reinforced polyester plastic panels offer high strength, corrosion resistance and low cost. Panels are well suited for chemical plant construction. Good indication of corrosion resistance is given in table showing results of 7-day immersion test in various corrosives.—INCO. 16782

#### 6.6.8, 5.4.3

**How Plastics Curb Corrosion in Metal Processing.** J. H. Lux. Iron Age, **179**, No. 3, 80-82 (1957) Jan. 17.

Lux lists plastic resins developed for heavy-duty corrosion-resistance. The use of plastic linings and acid-resistant ducts in cleaning, pickling, etching and plating operations is described.—MA. 15980

6.6.8, 8.1.2  
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**6.6.8, 8.1.2**  
**Silicones for Protection of Buildings.** (In German.) E. Pirson. *Kunststoffe*, 47, No. 10, 597-600 (1957).

Properties of silicones of importance in building protection, forms in which they are supplied and durability of treatment, are discussed.—RPI. 17896

## 7. EQUIPMENT

### 7.3 Pumps, Compressors, Propellers and Impellers

**7.3**  
**Right Material Cuts Pump Corrosion.** F. R. Drahos, Byron Jackson Company. *Chemical Engineering*, 65, No. 5, 162, 164, 166 (1958) March 10.

Outlines forms of corrosion encountered in centrifugal pumps, causes thereof, and prevention by selecting optimum material of construction. Galvanic, uniform, erosion, intergranular, pitting and cavitation erosion forms of corrosion are described. Economics of material selection are considered, and common pump materials (including stainless steels and nickel-based alloys) are reviewed. Photographs show examples of erosion pitting and cavitation corrosion.—INCO. 15464

**7.3, 4.6.11, 3.5.3, 6.3.6**  
**Corrosion and Erosion of Screw Propellers.** (In Japanese.) M. Kanamori and S. Ueda. *Metals*, 28, 647-651 (1958) Sept.

Manganese bronze and nickel-aluminum bronze are mainly used. Corrosion of screw propellers in sea water is caused by an electrolytic reaction in weak alkali solution in which oxygen is dissolved, and ionized zinc is removed from manganese bronze (or aluminum is removed from nickel-aluminum bronze). Cavitation erosion is caused by an impulse which is at most 200 kg per sq mm. Furthermore, screw exerts torque, friction and centrifugal force, which exert tension, bending, torsion and compressive stress, and hence mechanical strength is necessary.—MR. 17369

**7.3, 6.2.5, 8.8.3**  
**Cast Stainless Pump Handles Corrosive Electrolyte.** E. A. Schoefer. Alloy Casting Institute. *Plating*, 45, No. 4, 366-367 (1958) April; *Elec. Mfg.*, 62, No. 1, 122 (1958) July.

In continuous heavy copper plating of steel wire at Point Breeze Works of Western Electric Co., standardization heavy duty sump pumps are used to handle corrosive copper fluoborate electrolyte. All pump parts in contact with electrolyte are made of type CN-7M and type CF-8M. Most of the numerous cast stainless parts, such as impeller and impeller housing are statically cast; supporting and discharge pipes are centrifugally cast. In final leg of set up, CN-7M alloy pumps handle hydrofluosilic acid electrolyte used in applying lead plate.—INCO. 16662

### 7.4 Heat Exchangers

**7.4.1**  
**Use Salt Water Cooling Towers.** W. W. Mitchell. Standard Oil Co. of Calif. *Chem. Eng.*, 65, No. 12, 181-182 (1958) June 16.

California Oil Co.'s refinery at Perth Amboy is planning a salt water cooling tower. Tower will consist of induced draft counter-flow redwood cooling

tower cells similar to fresh water towers except for modifications to reduce drift and some differences in materials. Experience with original tower, which had silicon bronze interior hardware, led to use of all Monel hardware within tower. Fan blades and laminated disk assemblies of drive coupling are Monel. All pumps will have stainless steel cases and impellers and Monel shafts. All cooling water lines will be cement-lined steel pipe.—INCO. 16958

#### 7.4.1, 3.3.2, 8.4.3

**How 31 Refineries Condition Cooling-Water Systems.** J. D. Helwig and H. F. McEconomy. Atlantic Refining Co. *Oil and Gas J.*, 55, No. 48, 101, 103-106 (1957) Dec. 2.

Survey of oil industry's cooling water situation on condition methods employed in open circulating-type systems supplied by fresh makeup water showed that fouling and corrosion occur in all refinery cooling-water systems when treatment is not practiced. In some systems fouling is very heavy and corrosion rates may be as much as 0.3 to 0.4 in. per year. Problem is actively and effectively being combatted by chemical treatments, cathodic protection and special materials of construction. Each cooling-water system is a separate problem and requires a tailor-made treatment for optimum protection. Use of polyphosphates and organic inhibitors for cooling-water treatment dominates test programs conducted at various refineries. Tables.—INCO. 16189

#### 7.4.1, 4.3.7

**Redwood Cooling Tower Resists Corrosive Black Liquor Gases.** G. Weyermann and F. C. Ratliff. Crown Zellerbach Corp. *Chem. Processing*, 20, No. 8, 180-181 (1957) Aug.

Performance of redwood cooling tower handling corrosive sulfate process evaporator cooling water, has been excellent. This tower at Gaylord Container Corp., Bagolusa plant, has stainless steel pumps, piping, nails and bolts.—INCO. 16398

#### 7.4.1, 4.6.4, 3.2.2

**A Contribution to the Explanation of Corrosion in Cooling Tubes.** (In German.) A. Wyszomirski, H. Rudiger, E. Bachran and O. Damm. *Energietechnik*, 7, No. 9, 403-412 (1957) Sept.

Sheet-like corrosion and pitting of water tubes in industry. Properties of the cooling water used in testing water between 50 to 60 C. during 248 days. Incrustation.—BTR. 15142

#### 7.4.2, 5.2.1

**Corrosion Phenomena of Tubular Condensers. Characteristic Examples. Prevention and Correction.** (In French.) A. J. Maurin. *Corrosion et Anti-corrosion*, 5, No. 10, 275-285 (1957) October.

Description and use of a tubular condenser. Cathodic protection. Chlorination. General rules to prevent corrosion.—BTR. 15382

#### 7.4.2, 4.6.4, 4.6.7, 3.2.2

**Corrosion of Cooling Equipment in Refining.** (In French.) Marcel Prillieux. *Corrosion et Anti-Corrosion*, 5, 128-130 (1957) April.

Condenser failure in important units due to deep corrosive pitting by water has led to the investigation of the causes of this phenomenon. It seems that this corrosion is due to the weak oxygen content dissolved in the river water used for cooling.—MR. 15912

### 7.7 Electrical, Telephone and Radio

**7.7**  
**Fast-Heating Electronic Systems.** K. S. Hardin. Sandia Corp. *Electronic Equipment Eng.*, 6, No. 7, 39-42 (1958) July.

Discusses circuitry and tube design for achieving system that becomes operational in 10-12 seconds after heater power is applied. In tube design, active nickel alloys such as A-31, which contain various amounts of reducing agents, are generally used for cathode bases. Characteristics of fast-heating tubes are charted; cathodes are of Inconel, Cathalloy, and nickel.—INCO. 17330

#### 7.7, 4.2.4, 4.2.5

**A Field Study of ACSR Cable in Severe Marine and Industrial Environments.** E. W. Greenfield and E. W. Everhart. *Power Apparatus and Systems*, No. 29, 106-117 (1957) April.

Changes in electrical, mechanical, and chemical properties as caused by various climatological conditions are reported.—BTR. 14634

**7.7, 2.3.5, 4.5.2**  
**Corrosion of Electric Units and Other Objects in Soil by the Action of Electric Current.** (In Serbian.) Pance Kirkov. *Zastita Materijala*, 5, No. 1, 5-8 (1957).

Rate of corrosion of iron, zinc, copper, tin, lead, copper + zinc (40 percent zinc), platinum in the soil, conducted in a laboratory unit, in which samples were inserted in a vessel containing sand that had been treated with hydrochloric acid and were polarized with alternating

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ing current. Alternating current affects to a considerable extent the rate of corrosion, depending on the duration and the manner of introducing the current into the system.—MR. 17920

#### 7.7. 6.3.8

**A Lead-Tin-Barium Alloy for the Positive Grids of Lead/Acid Batteries.** N. L. Parr, A. Muscott and A. J. Crocker. *J. Inst. Metals*, **87**, No. 10, 321-329 (1958-59).

A metallurgical examination of the deterioration of lead alloy grids of acid batteries during service life revealed the factors to be considered during new alloy development. A lead-tin-barium alloy in laboratory trials gave the combination of castability, stiffness, creep strength and resistance to strong anodic stress-corrosion demanded by manufacture and service. (auth)—MA. 18010

#### 7.7. 6.4.2

**Aluminum Alloy Busbars.** F. McDonald. *Australasian Engr.*, 1958, 73-76, May 7.

In D50S the aluminum industry offers a new alloy specifically designed to meet busbar requirements, where both high strength and high conductivity are necessary. Busbars from this magnesium silicide-containing aluminum alloy can be made mechanically, as well as electrically, equivalent to their copper counterparts, and with cost savings of 50 per cent. Table presents properties.—INCO. 16550

## 8. INDUSTRIES

### 8.4 Group 4

#### 8.4.1

**Causes and Prevention of Corrosion in Tar Stills.** D. McNeil. *Corrosion Technology*, **4**, No. 11, 385-389 (1957) Nov.

Reviews experience over past 25 years with corrosion in coal tar stills, and presents results of recent investigations carried out by Coal Tar Research Association. Tests covered variety of relevant topics such as distribution of ammonium chloride in distillate oils and residual pitch during pot-still distillation, comparative corrosive effects of inorganic compounds in tar, effect of alkali addition on both corrosion of mild steel by hot tar and on distribution of ammonium chloride in tar distillate oils, corrosive properties of various distillate oils and of water-soluble and caustic soda-soluble components of these oils and effect of different metals (including Ni-Resist, Inconel, stainless steels FV 254, F.M.B. and F.D.P., mild steel, cast iron, Micro-sil and aluminum alloys.) Most tests were performed on specimens of mild steel containing 0.05 nickel. Tests showed that agents corrosive to mild steel in crude coal tar are inorganic salts dissolved in liquor associated with tar and phenolic compounds boiling above 240°C. Of inorganic constituents, ammonium chloride is the most aggressive. For construction of lower part of fractionating column, order of suitability for condenser material is stainless steel and chromized mild steel, followed by Inconel, cast iron, mild steel, and aluminum (poorest). For construction of flash chamber, cast iron is five times as resistant as mild steel; Inconel and Micro-sil are more resistant and stainless steels are virtually unattacked. Tables, graphs.—INCO. 15357

#### 8.4.3, 3.4.8, 4.3.3

**Does Higher Fuel Efficiency Increase Corrosion?** N. Seely. *Corrosion Technology*, **5**, No. 11, 355-356 (1958) November.

As thermal efficiency of modern high pressure, high temperature oil burning plant has increased, there has been similar increase in corrosion. Extent of problem is due to presence of vanadium, sodium and sulfur in currently available residual fuel oils. V<sub>2</sub>O<sub>5</sub> melts at 650°C and at higher temperatures attacks even highly alloyed steels. Possibility of adding neutralizing agent which reacts with V<sub>2</sub>O<sub>5</sub> to form chemically inert compound is investigated. Basic metal oxides and carbonates may be used for this purpose. Difficulty, however, lies in making a homogeneous addition of these powdered components. Author describes a colloidal suspension, Slipco, which includes chemically pure Ca(OH)<sub>2</sub> and zinc oxide stabilized with inorganic calcium compounds, together with other compounds. This agent prevents vanadium corrosion and also inhibits formation of SO<sub>3</sub> and sulfuric acid which form from relatively harmless SO<sub>2</sub>.—INCO. 17829

#### 8.4.3, 3.4.8, 3.2.3

**Regenerative Platinum-Catalyst Reforming.** H. C. Nix. *Humble Oil & Refining Co. Paper before American Petroleum Institute, Div. of Refining, Philadelphia, May 16, 1957. Proc. Am. Petrol. Inst.*, **37**, Sect. III, 380-385 (1957); *Oil & Gas J.*, **55**, No. 20, 168-170, 172 (1957) May 20.

Description of regenerative platinum catalyst reforming method at Humble Oil & Refining Company for producing high-octane components required in blending modern premium gasoline. Principal difficulties encountered in operation were caused by sulfur and hydrogen sulfide corrosion of hydrocarbon piping, which resulted in metal loss and scale formation. Sulfur corrosion rates of 200 mils per year were experienced in carbon-1/2 molybdenum tubes of naphtha preheat furnace. Hydrogen sulfide corrosion rates of 60-90 mils per year were experienced in low chromium-molybdenum reheat tubes and transfer units. Furnace was retubed with 9 chromium-1 molybdenum tubes. No corrosion was found in 18-8 stainless in the converted reformer. Tables, graphs, diagram.—INCO. 17966

#### 8.4.3, 3.2.2, 3.5.8, 6.2.5, 6.3.3

**Some Typical Cases of Corrosion in Platforming Units.** D. J. Bergman and S. A. Bjorkman. *Paper before Am. Petroleum Inst., Div. Refining, 22nd Midyear Mtg., Philadelphia, May 13, 1957. Proc. Am. Petroleum Inst.*, **37**, Sect. III, 93-99 (1957).

Discusses cases of corrosion encountered in Universal Oil Products Co. platforming units at several locations. Particular emphasis is placed on high chromium-nickel rod heater tube corrosion, intergranular corrosion of stainless steel valve trim in cold zones and stress corrosion of Monel impellers in centrifugal recycle-gas compressors. Photos, graphs, tables.—INCO. 17868

#### 8.4.5, 6.2.5, 6.3.15, 6.3.20, 3.2.2

**Summary of Specimen Corrosion Data from Slurry Blanket Mock-Up Run SM-3.** S. A. Reed and E. L. Compere. *Oak Ridge National Lab., U. S. Atomic Energy Comm. Pubn., CF-57-10-5, Oct.*

tober 10, 1957, 16 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C.

Examination of corrosion specimens from the 948 hour slurry blanket mock-up Run SM-3 indicated generally low attack rates on type 347 stainless steel, titanium-75A and Zircaloy-2. The upstream coupon of the array, of type 347 stainless steel, suffered moderate but more severe attack. Coupons of SA-212 grade boron carbon steel were severely attacked. No stress corrosion cracking was noted on samples, placed in the pressurizer, of types 347 and AM 350 stainless steels, titanium-aluminum-vanadium alloy or Zircaloy-2. (auth)—NSA. 17987

#### 8.4.5

**Recovery of Enriched Uranium from Uranium Dioxide-Stainless Steel Fuel Elements by Solvent Extraction.** J. R. Flanary and J. H. Goode. *Oak Ridge National Lab., Tennessee, Ind. and Eng. Chem.*, **51**, 23-26 (1959) Jan.

Laboratory studies of the batch Darex dissolution process, as applied to prototype APPR fuel, followed by continuous solvent extraction have yielded chemical flowsheets which provide excellent uranium recovery and decontamination from fission products and inert impurities. The advantages of the process include rapid dissolution of stainless steel-containing fuels, hydrogen-free off-gas, and relatively high throughput. The principal disadvantages are corrosion and the carry-through of inert metals with limited solubility to the radioactive waste storage system. (auth)—NSA. 17703

#### 8.4.5, 3.2.3, 6.2.5, 6.3.20, 6.3.15

**HRE-2 Corrosion Specimens—Blanket Region of Pressure Vessel (Loading No. 1)—Weight Data and Scale Analysis.** A. R. Olsen. *Oak Ridge National Lab., U. S. Atomic Energy Commission Pubn., CF-58-10-83, October 31, 1958, 13 pp. Available from Office of Technical Services, Washington, D. C.*

Specimens (type 347 stainless steel, zirconium alloys, and titanium alloys), exposed in the Blanket region of the HRE-2 during runs 13 and 14 as well as during some prior high temperature operations, were examined and weighed. The visual observations and weight data are presented together with the chemical analyses of scale removed from some of these specimens. The evidence indicates that the corrosion of the specimens was moderate. Some possible interpretation of the results of the scale analyses as related to core tank corrosion are also reported. (auth)—NSA. 17541

#### 8.4.5

**Experimental Determination of the Radiation Decomposition of Boron-10 Enriched Boron Trifluoride and the Resultant Corrosion of the Containing Materials.** W. K. McCarty. *Atomics International Div., North American Aviation, U. S. Atomic Energy Commission Pubn., NAA-SR-1999, October 15, 1957, 32 pp. Available from Office of Technical Services, Washington, D. C.*

A series of experiments is described in which aluminum capsules containing metal specimens were filled with B<sup>10</sup> enriched boron trifluoride and irradiated in the MTR. Post-irradiation measurements determined the extent of radiolysis of the BF<sub>3</sub> and the extent of corrosion of the metal specimens. Neither of these phenomena was found to be a

serious problem in the devices investigated. (auth)—NSA.

8.4.5 **Corrosion of Nuclear Reactor Components.** M. Jaeger. *Mechanical Problems of the Nuclear Power Plant*, 505 (1957).

8.4.5, 3.2.2 **Corrosion Ions on Aerial Electric Products.** Open Commission, 17, 1953 (1) 6 pp. Available from Service, V.

The commissioning water heavy metal investigation per impurity on corrosion—NSA.

8.4.5 **The Effect of Frequency Uncertainty Rate on National Commission, 16, 1953 (1) 7 pp. Available from Services, Revision of Corrosion experiments—NSA.**

8.5 **Group 5**

8.5.3 **New Continuous Process.** Ind., 40, 1 pp. Described Corp's plant in Alabama. Al liquor in stainless steel recovered, turpentine a spirit.

Type 304 stainless steel equipped and pulped to constant tension made of molybdenum.

8.5.3, 3.2.2 **Control Magnesium Ketone, TAPPI, Oregon, No. 24, No. 2.** Type 316 mill and fabricated closed systems which resist corrosion and expandable specifications.

serious problem in the design of safety devices incorporating boron trifluoride. (auth)—NSA. 15339

**8.4.5 Corrosion in Primary Cooling System of Nuclear Reactors.** (In German.) T. Jaeger, *Maschinenbau Technik*, 6, 495-505 (1957) Sept.

Problems caused by the induced activation of impurities in the cooling medium used in nuclear reactors. 27 references.—MR. 16228

**8.4.5, 3.2.2, 3.4.8, 6.4.2 Corrosive Effects of Heavy Metal Ions on Aluminum.** G. S. Fujioka, General Electric Co., Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-27434, March 17, 1953 (Declassified Feb. 20, 1957), 12 pp. Available from Office of Technical Service, Washington, D. C.

The corrosive effects of reactor cooling water which contained traces of heavy metal ions on aluminum were investigated. Results indicated that copper impurities contributed to the pitting corrosion of aluminum.—NSA. 14924

**8.4.5 The Effect of Run Duration, Sampling Frequency, and Nickel Pickup on the Uncertainty in Observed HRE Corrosion Rates.** E. L. Compere, Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-53-7-100, July 16, 1953 (Declassified February 14, 1957), 7 pp. Available from Office of Technical Services, Washington, D. C.

Revisions in the proposed HRE corrosion experiments are briefly discussed.—NSA. 15223

## 8.5 Group 5

### 8.5.3

**New Gulf States' Pulp Mill Uses Continuous Process.** A. W. J. Dyck, Paper Ind., 40, No. 1, 16-23, 32 (1958) April. Description of Gulf States Paper Corp's pulp mill at Demopolis, Alabama. All metal parts in contact with liquor in heat exchangers are cast 18-8 stainless steel and wrought Type 304 stainless. Turpentine and relief gases are recovered from presteaming vessel by a turpentine recovery system incorporating a spiral heat exchanger made of Type 304. Pipes throughout bleach plant are either rubber lined or made of stainless steel. Pulper of pulp machine is equipped with a tank made of stainless and pulping unit is of all chromium steel construction. Black liquor is pumped to evaporators by corrosion resistant Type CNG chemical pumps made of high nickel, high chromium-molybdenum alloy. Photos.—INCO. 16481

**8.5.3, 3.2.2, 4.3.2 Controlling Corrosion in a Modern Magnesia-Based Sulfite Mill.** J. E. Finsen, Ketchikan Pulp Co. Paper before TAPPI, 13th Eng. Conf., Portland, Oregon, July 28-August 1, 1958. TAPPI, 42, No. 2, 104-108 (1959) Feb.

Type 316 did not perform according to expectations in magnesium bisulfite mill and premature failures occurred in fabricated piping and equipment. With closed system for recovery of chemicals, injurious chlorides were not removed as expected; instead they become concentrated to critical levels. Paper outlines forms of corrosion encountered and experiences in control of corrosion which resulted in adoption of rigid specifications for material quality, design

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and fabrication procedures. Pitting experienced in heat exchanger tubes and valve castings was related to wide range of molybdenum contents of stainless steel. Pit corrosion has been reduced to negligible proportions by adopting minimum of 2.75 molybdenum. Stress corrosion cracking occurred in acid and liquor piping where frequent and abrupt temperature changes occur, and in locations where hydraulic pulsations set up vibration in piping; at present stress is only variable that can be controlled if chlorides cannot be eliminated. Problem of intergranular corrosion is covered and proper heat treatment and welding techniques to limit sensitizing are discussed. Importance of design to prevent concentration cell or crevice corrosion is stressed. Splashing effect or hot wall corrosion is considered briefly.—INCO. 17794

**8.5.3, 4.3.3, 6.3.3 Corrosion of Steel in Alkaline Pulping Liquors.** E. Uusitolo. (In Finnish.) Paper ja Puu, 40, 393-397, 443-446 (1958); Paper Ind., 40, No. 9, 630 (1958) Dec.

Study of liquor components that promote marked corrosion in kraft cooking liquors. Attempts were made to determine conditions under which corrosion could be reduced to a minimum. Small amounts of polysulfides actually promoted corrosion, but the addition of larger amounts accelerated the transformation of the steel to a passive state

and thus arrested corrosion. It was shown that liquors that contain 100 g of caustic soda and 35 g of sodium hydrogen sulfide per liter were high enough to insure marked digester corrosion under certain conditions. Such corrosion could be prevented effectively by a slight reduction of the caustic soda content at the beginning of the digestion, and corrosion could be prevented almost completely by addition of polysulfides to the liquor in amounts that varied depending on the cooking conditions. Graphs.—INCO. 17641

**8.5.3, 4.3.3, 6.2.2 Manufacture of Cold Soda Pulp from Eucalypt Woods.** E. J. Bugg and A. J. Pearson. Paper Trade J., 142, No. 51, 18-24 (1958) Dec. 22.

Report on pioneering work on production of ground wood type pulp for newsprint using hardwood chips and a mild cold caustic treatment without pressure. All refiner parts in contact with pulp or process liquors are of Type 304 stainless steel. Feed screw, hopper and all splash guards and covers are of Type 304, drain trough is metallized inside with Type 316 while all barrel rings, clamping bars, pressure bars and keeper knife bars are chemically plated with Alecoplate containing 93 nickel. Use of Ni Hard refiner plates has reduced the overall plate cost from corrosion loss from 15/8 to 3/2 per ton of pulp. Tables, diagrams.—INCO. 18021

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**T-2H-5 To Review and Encourage Publication of Technical Articles on Asphalt-Type Protective Coatings**

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(Chairman to be Selected)

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## Topic of the Month

### Stress Cracking of Polyethylene Cable Insulation

By H. HOWARD BENNETT\*

#### Introduction

THE RAPID and unexplained failure of polyethylene insulated cable was experienced on a cathodic protection installation on two steel tanks in steam generator feedwater service. Six non-sacrificial type anodes were installed in each tank to provide proper current distribution. They were suspended from the roof with insulated cable to locate them vertically at the middle of the liquid for normal tank level. The average temperature of the water was approximately 125 F.; however, under abnormal conditions it is possible for it to reach 200 F.

Slight traces of oil appeared at times in the returned condensate. This oil was removed periodically with the overflow. For these conditions, especially the high temperature, high molecular weight polyethylene was chosen as insulation for the cables used to suspend the anodes from the tank roof.

After a few months of operation, erratic current readings at the rectifier led to an examination of the anode system. It was found that the polyethylene cable insulation had failed by cracking, as shown in Figure 1. The failure exposed the stranded copper cable to the water, making it part of the anode system. This was undesirable because copper is rapidly attacked as an anode and copper salts would be introduced into the feedwater.

Further study of the cracked insulation revealed that it had absorbed some oil, but no apparent swelling had taken place. The polyethylene was firm and flexing did not cause further cracking. Abrading the surface indicated that the oil had penetrated sufficiently to cause surface deterioration.

#### Effect of Oils

A literature review<sup>1,2</sup> disclosed that polyethylene is subject to stress cracking in the presence of certain solvents and oils. Howard<sup>2</sup> defines this phenomenon as follows: "Environmental stress-cracking is the failure in surface-initiated brittle fracture of a polyethylene specimen or part under polyaxial stress in contact with a medium in the absence of which fracture does not occur under

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the same conditions of stress. Combinations of external and/or internal stress may be involved, and the sensitizing medium may be gaseous, liquid, semi-solid, or solid."

The materials that cause cracking degrade the surface of the plastic sufficiently to initiate cracks under conditions of stress and elevated temperature. Apparently, in this case the presence of small traces of oil, stress and temperature produced the right conditions to cause stress cracking in a very short time.

Other plastics for use in this service are being studied. Among them is polypropylene which has adequate tempera-

#### Abstract

A case history is given of the failure of polyethylene cable insulation in a cathodic protection installation using non-sacrificial anodes. The cathodic protection installation was on two steel tanks in steam generator feedwater service. It was decided that the presence of small traces of oil, stress and temperature produced the right conditions to cause the stress cracking of the insulation. 5.2.1

ture resistance and is not subject to stress cracking. It is planned to place this material on test.

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1. H. E. Atkinson, Plastic Piping Systems, *Chem. Eng. Progress*, (1959) November.
2. J. B. O. Howard, A Review of Stress-Cracking in Polyethylene, Paper—SPE 15th Annual Tech. Conf., N. Y. C. (January, 1959).

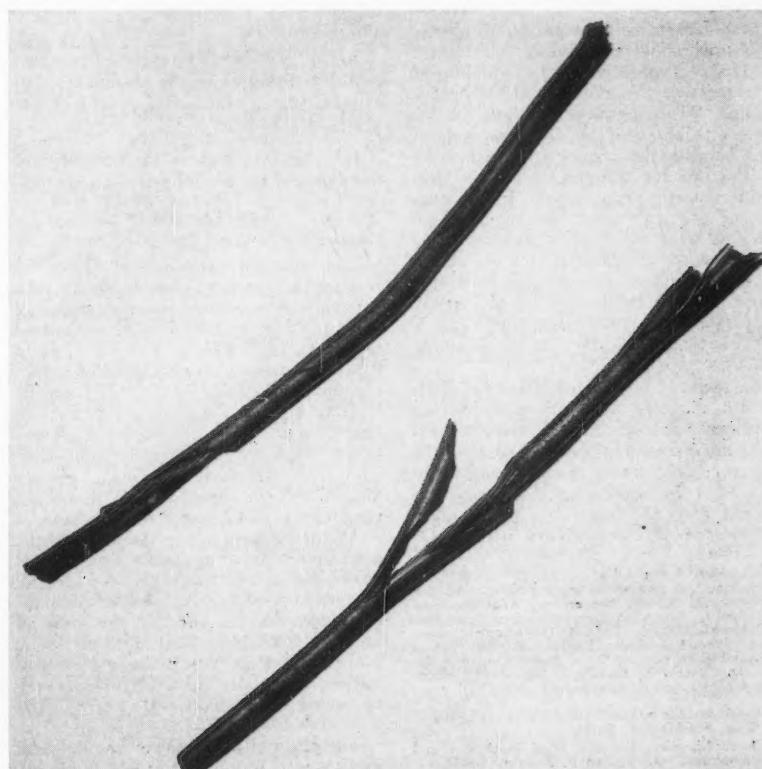


Figure 1—Cracked polyethylene insulation.

# Materials of Construction for Equipment Used With Hydrogen Peroxide\*

By RALPH BLOOM, JR.,<sup>(1)</sup> LORNE E. WEEKS<sup>(2)</sup> and CHARLES W. RALEIGH<sup>(3)</sup>

## Introduction

**A**NHYDROUS hydrogen peroxide and hydrogen peroxide aqueous solutions act as oxidants, reagents, solvents, gas generators and energy producers. They are susceptible to heterogeneous catalytic decomposition, especially in contact with heavy metals and their oxides. When completely isolated from their environment, pure or stabilized  $H_2O_2$  solutions are extremely stable. However, because of their reactivity, care must be exerted in the selection of the materials of construction in which they are to be handled and stored.

The Becco Chemical Division, Food Machinery and Chemical Corporation, has evaluated materials of construction for  $H_2O_2$  service throughout its 33 year manufacturing history. The purpose of this paper is to summarize the subject of materials of construction for equipment for handling  $H_2O_2$  solutions of all concentrations from the diluted 3-6 percent drug store and beauty shop product to the 90-99 percent propellant product.

For the initial survey evaluation program for 90 percent  $H_2O_2$ , the use of accelerated compatibility testing was selected. Therefore, test procedures were chosen which were quantitative in respect to  $H_2O_2$  decomposition, but qualitative in respect to the effect of  $H_2O_2$  on the sample. Then, because the bulk of the data was obtained by these procedures and because the results appeared to be satisfactory for the purpose, these procedures were perpetuated. In general, use of the procedures described herein has led to a satisfactory experience in the selection of materials for all phases of  $H_2O_2$  service. This success may in a large measure be due to the philosophy that laboratory tests are only a guide and that the real test lies in obtaining actual service experience.

In approaching the selection of materials for a special application, it must be considered that the compatibility test results are general in nature and must be interpreted in terms of the specific conditions of the application. Naturally, one should select the material with the highest degree of compatibility with  $H_2O_2$ .

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## Abstract

Materials of construction are evaluated in 90 percent by weight hydrogen peroxide. Topics discussed include factors requiring materials evaluation, test procedures, service classifications for materials, criteria for evaluating test results, results of compatibility evaluations, preparation of materials for hydrogen peroxide service, test results and service experience, design and fabrication techniques recommended for hydrogen peroxide storage and handling equipment, and problem areas encountered. Test results are reported on the following materials: pure metals, aluminum and its alloys, stainless steel, miscellaneous alloys, polyethylene and halogenated polyethylenes, PVC and copolymers, silicone rubbers, miscellaneous plastics, synthetic oils and greases, and miscellaneous materials. Data reported include compatibility tests with 90 percent hydrogen peroxide at 66°C on metals, plastics and lubricants. Compatibility of materials with 98 percent hydrogen peroxide at 66°C is included. 43.7

blistering, surface disintegration, etc. Weight or dimension change measurements were not included.

## Stability Tests

As indicated above, the evaluation of the compatibility of materials with hydrogen peroxide involves evaluation of both the effect of the material on the  $H_2O_2$  and of the  $H_2O_2$  on the material. Therefore, the test procedures are designed to evaluate the following:

1. The quantitative effect of the material on the rate of decomposition and the stability of the  $H_2O_2$ .
2. The qualitative effect of the  $H_2O_2$  on the material itself such as corrosion of metals by either the liquid or vapor, and the hardening, swelling, blistering, deterioration, dissolving, etc. of plastics and elastomers.
3. The possible formation of impact sensitive mixtures of the materials or of components of the material with  $H_2O_2$  during contact.
4. In special instances, the suitability of the material for the intended application.

## Test Procedures

### Compatibility

The effect of materials on  $H_2O_2$  decomposition and of the  $H_2O_2$  on the materials is evaluated by immersion of the sample in the  $H_2O_2$  with a standard wetted surface-to- $H_2O_2$  volume ratio of 0.33 sq inch/cu inch at controlled standard temperatures of 30°C (86°F) and 66°C (151°F) and for standard periods of time of four weeks and one week respectively. This test is generally applied to solid materials such as metals, plastics and elastomeric compounds. With a slight variation, it can be applied to greases.

All the surfaces of the samples and the glassware were scrupulously cleaned and passivated before materials were tested. The results of the immersion compatibility tests for the effect of the material on the rate of decomposition of the  $H_2O_2$  are reported as the percent of active oxygen loss (percent AOL) from the contacted  $H_2O_2$  per week at 66°C (151°F) and 30°C (86°F).

The effect of the  $H_2O_2$  on the material itself was determined visually, with notations made of corrosion, staining, bronzing, pitting, swelling, bleaching,

## Impact Sensitivity Tests

Liquid and powdered materials (including solids which might yield finely divided particles in service) must be evaluated for the possible sensitivity to impact when in intimate contact with  $H_2O_2$  in various proportions. Impact sensitivity is determined by subjecting varying proportions of the material and  $H_2O_2$  to the impact of a weight dropped from a specified height. In early tests a 1 kilogram meter (7 ft-lbs.) impact was used. Later a 3 kg-meter (21 ft-lbs.) was provided. Now impacts up to 100 ft-lbs. (14.6 kg-m) may be attained. Most of the results reported were from tests at 1 and 3 kg-m impact. The apparatus used for this test is a modified Bureau of Mines Liquid Explosive Impact Tester which may be used for testing at various temperatures up to 270°F.

## Special Material Tests

In general, the suitability of the material for the intended application is indicated in the laboratory tests. For example, an elastomer which becomes brittle during immersion in  $H_2O_2$  is not suitable for the fabrication of "O" rings for  $H_2O_2$  service. However, some materials cannot be adequately evaluated in such tests. This is particularly true of coatings, protective clothing, and joint sealing compounds; special procedures have been developed for testing these particular materials.

## Experience

The final evaluation of a material for its suitability for an application involving contact with  $H_2O_2$  is on the basis of experience during use. In general, the recommendations of materials classification for  $H_2O_2$  service are based on the

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**TABLE 1—Criteria for Classification of Materials for H<sub>2</sub>O<sub>2</sub> Service on the Basis of Laboratory Tests**

Class	Material	Max. Amt. of Active Oxygen Loss/Wk.		Min. Stab. of H <sub>2</sub> O <sub>2</sub> After Test	Remarks
		30C	66C		
1...	Metals	0.2	5.0	95%	No other effect on H <sub>2</sub> O <sub>2</sub> or material.
1...	Plastics for drums and liners	0.2	5.0	95%	No other effect on H <sub>2</sub> O <sub>2</sub> or material and non-impact sensitive to 3 kg-meter impact at 212 F.
1...	Plastics for gaskets, tubing, etc.	0.4	6.0	95%	No other effect on H <sub>2</sub> O <sub>2</sub> or material and non-impact sensitive to 3 kg-meter impact at 212 F.
1...	Oils and greases	1.0	10.0	95%	No other effect on H <sub>2</sub> O <sub>2</sub> or material and non-impact sensitive to 3 kg-meter impact at 212 F.
2...	Metals	6.0	80.0	90%	No other effect on H <sub>2</sub> O <sub>2</sub> . Slight bronzing of the metal allowable but no corrosion.
2...	Plastics	6.0	80.0	90%	No other effect on H <sub>2</sub> O <sub>2</sub> . Slight bleaching, swelling, embrittlement or occasional blistering accepted. Non-impact sensitive to 3kg-meter impact at 212 F.
2...	Elastomers	6.5	95.0	90%	No other effect on H <sub>2</sub> O <sub>2</sub> . Slight bleaching or loss of small amount of elasticity of the material accepted. Non-impact sensitive to 3 kg-meter impact at 212 F.
2...	Oils and greases	6.0	80.0	90%	No other effect on H <sub>2</sub> O <sub>2</sub> . Non-impact sensitive to 3 kg-meter impact at 212 F.
3...	Metals	11.0	100/24 hrs.	15% <sup>1</sup>	Bronzing and staining, but not rusting or other corrosion products. Slight attack may be allowed.
3...	Plastics	11.0	100/24 hrs.	15% <sup>1</sup>	May become partially bleached, distorted, disintegrated, after 1 wk. on test. Non-impact sensitive to 3 kg-meter impact at 212 F.
3...	Oil and greases	11.0	100/24 hrs.	15% <sup>1</sup>	Non-impact sensitive to 3 kg-meter impact at 212 F. Excessive H <sub>2</sub> O <sub>2</sub> decomposition approaching catalytic rate.
4...	Metals	....	....	....	Metals: pitted and corroded during or after test.
	Plastics	....	....	....	Plastics: disintegrated, burned, blistered. Impact sensitive to 3 Kg-meter at 212F.
	Elastomers	....	....	....	Elastomers: burned, swollen, dissolved, disintegrated, blistered, gummy, loss of elasticity. Impact sensitive to 3Kg-meter at 212 F.
	Oil and greases	....	....	....	Oils and Greases: melted, disintegrated, burned. Impact sensitive to 3 kg-meter impact at 212F.

<sup>1</sup> After 30 C test.

results of laboratory tests and on practical experience. In a few instances, practical experience has shown results at variance with those of laboratory tests. Whenever this is the case, the greater consideration has been given to practical experience and conclusions drawn accordingly. Criteria established for laboratory tests are based as far as possible on correlations with experience resulting from placing the materials in service.

#### Service Classifications for Materials

The results of the laboratory evaluations and practical experience with various materials have shown that materials should be classified into categories based on their contemplated types of use. Thus all materials need not be suitable for indefinite storage because in applications requiring only short-time contact with the H<sub>2</sub>O<sub>2</sub>, materials of lesser degree of compatibility can be employed. To facilitate selection on this basis, materials have been classified according to the types of applications for which they are suited. A system of four classes has been developed which has been generally adopted for materials for hydrogen peroxide service.

#### Class 1—Materials Satisfactory for Unrestricted Use With H<sub>2</sub>O<sub>2</sub>

Such service includes long-time contact with the H<sub>2</sub>O<sub>2</sub>. Typical use: storage containers

#### Class 2—Materials Satisfactory for Repeated Short-Time Contact With H<sub>2</sub>O<sub>2</sub>

Such materials are used for:

- Transient contact with the H<sub>2</sub>O<sub>2</sub> prior to storage of the H<sub>2</sub>O<sub>2</sub> or
- Limited contact with the H<sub>2</sub>O<sub>2</sub> prior

to use. Such contact is not to exceed four hours at 160 F or one week at 70 F. Typical uses are (1) Valves and pumps in H<sub>2</sub>O<sub>2</sub> transfer lines, and (2) feed tanks

#### Class 3—Materials Which Should Be Used Only for Short-Time Contact With H<sub>2</sub>O<sub>2</sub>

This might be prior to a decomposition process or similar use, and should be used only where neither a Class 1 nor Class 2 material would suffice. These materials can be used for repeated contact, but no one period should exceed 1 minute at 160 F or 1 hour at 70 F prior to immediate use.

#### Class 4—Materials Not Recommended For Use With H<sub>2</sub>O<sub>2</sub>

These materials: (1) cause excessive decomposition of H<sub>2</sub>O<sub>2</sub> even on short-time contact, (2) are attacked or suffer deterioration on contact, (3) yield corrosion or deterioration products which cause excessive decomposition of H<sub>2</sub>O<sub>2</sub> on subsequent contact, or (4) form impact sensitive mixtures with concentrated H<sub>2</sub>O<sub>2</sub>.

#### Criteria for Evaluating Test Results

##### Standard Compatibility and Impact Tests

The present criteria limits for classifying materials for H<sub>2</sub>O<sub>2</sub> service on the basis of the tests outlined above and from experience in service are summarized in Table 1. Since the classification system is general for all construction materials for H<sub>2</sub>O<sub>2</sub> handling, the specific classifications are all inclusive. Limits for all classes were chosen arbitrarily. For specific applications, more precise limits may have to be established.

The application of the criteria of

Table 1 to the results of compatibility tests were subject to the following modifying considerations:

1. If there was any doubt as to whether a material should be in a given category, the material was placed in the lower category.

2. If the results of practical experience were at variance with the results of laboratory tests, then the greater weight was given to the practical experience in selecting the classification.

3. The main distinction between Class 2 and 3 was the possible effect on the stability of the H<sub>2</sub>O<sub>2</sub>. If there was any doubt as to whether the stability might be affected, the material was placed in Class 3. Slight deterioration of the material causing foreign matter to enter the H<sub>2</sub>O<sub>2</sub> might cause decreased stability of the H<sub>2</sub>O<sub>2</sub>.

4. Numerical limits for the various classes are approximate. Class 1 material would fall within rather narrow limits, and Class 2 material within much broader limits. In general, the higher the active oxygen loss for a particular material, the less reproducible are the results.

Any material which is impact sensitive when in contact with H<sub>2</sub>O<sub>2</sub> in any proportion is considered a Class 4 material. The criteria of impact sensitivity of a material in contact with H<sub>2</sub>O<sub>2</sub> is the incidence of any positive detonation on the basis of at least ten trials.

A mixture which gives negative results in ten tests is considered to be tentatively non-impact sensitive unless later tests produce a positive result.

### Results of Compatibility Evaluations

Representative results of compatibility tests which have yielded significant results are presented in Tables 2, 3, 4 and 5.

The data presented for each sample include all or part of the following:

1. Description and source.
2. Active oxygen loss by the  $H_2O_2$  in one week at 66°C (151°F). Only the 66°C results are reported because these are more significant and reliable than the 30°C results.

3. Stability of the  $H_2O_2$  remaining after stability test.

4. Results of impact sensitivity tests (where applicable).

5. Description of the samples after test.

6. Remarks about any unusual effect on the  $H_2O_2$  during the test.

7. Recommended classification.

All of the tests reported were carried out with 90 percent  $H_2O_2$  unless otherwise noted. Table 5 presents data obtained with 98 percent  $H_2O_2$ . Data applicable to concentrated  $H_2O_2$  is applicable to lower concentrations. However, the reverse is not always true.

### Preparation of Materials, Tanks and Equipment for $H_2O_2$ Service

To obtain satisfactory service from materials used for handling and storing hydrogen peroxide it is necessary to properly clean, passivate and condition these materials prior to such use. In general, chemical procedures have been employed throughout the industry, but these procedures have shown great variations in detail.

The procedures recommended by Becco on the basis of long experience consist of cleaning by means of a detergent solution, chlorinated solvent or a caustic rinse followed in the case of metals by passivation with nitric acid and a thorough, clean potable or distilled water rinse. Complete detailed procedures are available but space prevents their presentation here.

Tanks and equipment for concentrated hydrogen peroxide service, (i.e., 70 percent  $H_2O_2$  by weight or above), will yield best results if they are conditioned with commercial, stabilized, 130 volume, 35 percent by weight  $H_2O_2$  for at least 3-4 hours and preferably 24 hours at ambient temperatures. This "conditioning" period can also be considered a surveillance test period because observation of the  $H_2O_2$  should be made to determine excessive decomposition from the surface as a whole or from particular local spots. After the conditioning treatment, tanks and equipment must be thoroughly washed with clean potable or distilled water prior to use for handling concentrated  $H_2O_2$ .

Following preparation of tanks and equipment for  $H_2O_2$  service, an experienced operator can generally determine by visual observation whether or not the preparation has been effective. If there is no bubbling or gas streamer and the  $H_2O_2$  solution remains cool to the touch, indications are that the tank or item of equipment is satisfactory for  $H_2O_2$  service. During fabrication, care must be

taken to use practices that result in a minimum of defects.

### Discussion of Test Results and Service Experience

#### Aluminum and Aluminum Alloys

The results of a great number of tests indicate that there are only a very limited number of materials which meet the stringent criteria of Class 1 materials. Of these, controlled copper content pure aluminum, Aluminum Association Designation No. 1060, has been applied most extensively. Experience with 1060 aluminum for tanks, piping and equipment has been excellent; corrosion has been low. Most cases of corrosion are either local pitting, indicating possibly the presence of foreign matter, or more general attack which has been traced to the possibility of galvanic attack. Hydrogen peroxide containing a high chloride concentration will pit this aluminum as well as the aluminum alloys; however, 1060 aluminum appears to have greater resistance to corrosion than do the aluminum alloys. The addition of nitrate ion to the  $H_2O_2$  will tend to control chloride attack on aluminum.

Where higher mechanical strength is required of the metal for such uses as tank cars and transport truck tanks, aluminum alloys 5652 and 5254, both with copper content controlled at less than 0.06 percent by weight, may be used.

Tank cars of 4000 and 8000 gallon capacity fabricated from these alloys have been in satisfactory service with all concentrations of  $H_2O_2$  since 1952.

Another wrought alloy which has been employed in  $H_2O_2$  service is 6061, because of its high strength in the T-6 temper and because of its availability in bar stock form. Despite its satisfactory compatibility test results, 6061 is usually not recommended unless there is no other alloy available to meet the requirements. This alloy is more susceptible to corrosion in intermittent wetting applications than the other aluminum alloys used to handle  $H_2O_2$ ; therefore, sulfuric acid anodization followed by a one hour boiling water seal without dye is usually recommended if 6061 is to be employed for  $H_2O_2$  service. Type 6061 is not recommended for  $H_2O_2$  piping.

Wrought alloy 6363, which is a recent product with controlled copper content, showed excellent Class 2 compatibility with 90 percent  $H_2O_2$  in the laboratory tests; however, there is no use experience with this alloy to report at this time.

The high strength structural alloys such as 2014, 2017 and 2024 are unsuitable for service with  $H_2O_2$  because of corrosion and a high rate of  $H_2O_2$  decomposition in contact with them. Although sulfuric acid anodization improves the compatibility somewhat, Class 3 service is indicated by the laboratory results.

TABLE 2—Representative Results of Compatibility Tests With 90 Percent Hydrogen Peroxide—Metals

Material	Classification	Effect of Sample on $H_2O_2$		Apparent Effect of $H_2O_2$ on the Sample	Source of Material
		Percent Active Oxygen Loss per Week at 66°C	Stability After Test		
Wrought Aluminum		*			
1060	1	1.5	98.8	None	Alcoa
2014	4	100	...	None	Alcoa
3003	2	13.8	94.4	None	Alcoa
5652-0	2	5.0	98.4	None	Alcoa
5254-0	2	7.8	97.6	None	Alcoa
6061**	2	4.8	98.7	None**	Alcoa
6363	2	7.6	96.2	None	Alcoa
7072	1	2.1	...	None	Alcoa
Cast Aluminum					
356F	2	50			
B356	1	2.8	98.0	None	Alcoa
Chromic Acid Anodized 6061	3	15.5	...	None	Colonial Alloys
Sulfuric Acid Anodized 6061 (Detergent Wash)	2	4.5	90.8	None	Alcoa-(Keystone Chromium) Stolle Corp.
6061 Hard Coat .001" thick	3	23.2	64.2	None	
Stainless Steel					
202	2	19.0	89.0	Bronzed	Brace-Mueller
302	2	21.0	...	Bronzed	Whitehead
304	2	40.0	...	Bronzed	Allegheny-Ludlum
316	2	19.8	...	Bronzed	Republic
322	2	30.0	...	Bronzed	Carnegie
347	2	57	...	Bronzed	Carnegie
410	4	91.4	16.6	Rusted	Diesel Eng.
430	4	74.4	37.5	Rusted	Allegheny-Ludlum
17-7 PH, 37-45 Rc Special Pass.	2	20	...	Bronzed	Armco Steel
Carpenter 20	3	100	...	Bronzed	Carpenter Steel
Pure Metals					
Copper	4	100	0	None	
Iron	4	100	0	Rusted	
Lead	4	Catalytic	0	Dissolved	
Mercury	4	Catalytic	0	None	
Silicon	2	3.5	97	Slight surface dulling	General Electric
Silver	4	Catalytic	0	Attacked	
Tin-C.P.	2	28.7	...	None	Baker Chem. Co.
Titanium	4	100	0	Pitted	Rem-Cru Titanium Inc.
Zirconium	1	3.2	...	None	National Lead Co.

\* Percent AOL/week refers to percent Active Oxygen Loss by the  $H_2O_2$  in one week's test.

\*\* Field experience shows this alloy to be subject to corrosion.

Hypalon V  
Kel-F (unp.  
Kel F 370C

Kel F 550C  
Polyethylene

Teflon (wh.  
Viton A (S.

Koroseal I  
Koroseal II  
Koroseal 2A

Tygon B-7  
Tygon 3400  
Vinylite V  
Fluoroplastic

SE 450 (u.  
Silastic 15  
Silastic 6-  
Silastic 97

Silastic 97  
Silastic S-  
Silastic Y  
Buna N.

Butyl Ru.  
Hysol 4-7-  
Hysol 600  
Mylar B.

Neoprene  
Nylon  
Phenol-F  
Plexiglas

Polystyrene  
Thiokol E  
Thiokol 3  
Duroid 50

Fairprene  
Fairprene  
Korda FI  
Silastic L

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**TABLE 3—Representative Results of Compatibility Tests With 90 Percent Hydrogen Peroxide—Plastics**

Material	Classification	Effect of Sample on H <sub>2</sub> O <sub>2</sub>		Apparent Effect of H <sub>2</sub> O <sub>2</sub> on the Sample	Source of Material
		Percent Active Oxygen Loss Per Week at 66°C	Stability After Test		
Hypalon V-54-B (grey)	4	100	...	Blistered and swollen.	Raybestos-Manhattan
Kel-F (unplasticized)	1	3.5	...	None.	Minn. Mining & Mfg. Co.
Kel-F 800 (Lot 5649)	1	4.5	97.8	Very slight hardening.	Minn. Mining & Mfg. Co.
Kel F 3700 gum	3	8.8	93.2	Blistering and bleaching.	Minn. Mining & Mfg. Co.
Kel F 5500 gum	3	18.5	...	Blistered and distorted.	Minn. Mining & Mfg. Co.
Polyethylene	2	1.0	95	None at room temperature—can detonate with H <sub>2</sub> O <sub>2</sub> if heated.	DuPont
Teflon (white)	1	2.8	95	None.	DuPont
Viton A (411A4) (black)	3	80.6	82.1	Bleached to blue color, blistered and swelled.	Hewitt Robins
Koroseal 116	3	23	...	Bleached and hardened.	B.F. Goodrich
Koroseal 117 (molded)	3	16	...	Bleached and hardened.	B.F. Goodrich
Koroseal 700	2	2.0	...	Slightly swollen stiffened and bleached.	B.F. Goodrich
Saran	2	12.3	...	Bleached.	Dow Chemical Co.
Tygon B-20	3	2.3	...	Bleached, blistered, and distorted slightly.	U.S. Stoneware
Tygon 3400	4	...	...	Embrittled and swollen.	U.S. Stoneware
Vynilite VU 1930	3	2.4	...	Turned opaque, slight blisters.	Bakelite Div.
Fluorosilicone LS-53	2	16.7	91.3	None.	Dow Corning
SE 450 (unpigmented)	2	13.3	...	Very slight bleaching and swelling.	General Electric Co.
Silastic 152	3	17.8	68.1	Slightly bleached.	Dow Corning
Silastic 6-128	2	6.7	99.7	Bleached, very slightly blistered.	Dow Corning
Silastic 9711	2	7.4	99.1	Slightly bleached.	Hewitt Robins
Silastic 9711 welded with S-2200	2	13.6	96.0	Weld unaffected. Slight bleaching and mottling.	Dow Corning
Silastic S-2000-4-480	2	16.1	95.5	Bleached and mottled slightly.	Dow Corning
Silicone Y-1749	2	12.3	...	Small white blisters.	Carbide & Carbon Co.
Buna N	4	76.7	83.1	Dissolved.	
Butyl Rubber A 3405	4	100	...	Softened to sticky mass overnight.	Esso Lab.
Hysol 4-77C (clear)	4	100	...	Bleached, distorted.	Houghton Labs.
Hysol 6000B (amber)	4	100	...	Partially dissolved.	Houghton Labs.
Mylar B	1	1.7	...	None.	DuPont
Neoprene Pure Gum	4	...	...	Ignited and burned.	Spray Dip Co.
Nylon	4	...	...	Dissolved.	DuPont
Phenol-Formaldehyde	4	100	...	Sample burned.	Durez
Plexiglas	4	100	...	Softened and partial solution.	Rohm & Haas
Polystyrene (Polystyrene)	2	9.1	...	None.	Plax Corp.
Thiokol EC-801-LP2	4	100	0	Burst into flame.	Thiokol Corp.
Thiokol 3000 FA	4	100	...	Dissolved.	Thiokol Corp.
Durod 5600 (fiber-reinforced Teflon)	3	44.7	70.1	None.	Rogers Corp.
Fairprene PS57-168 (Viton A, Dacron)	2	23	94.5	Moderate surface attack.	DuPont
Fairprene 5809 (Viton A)	2	7.5	...	None.	DuPont
Korda Flex (Teflon coated glass fabric)	2	2.5	97	None.	Chicago Gasket Co.
Silastic DC-9711 on Dacron diaphragm	3	9.7	96.4	Blistered and distorted.	Kirkhill Rubber Co.

Aluminum casting alloys 43 and 356 have been employed successfully for pump and valve bodies for many years although some corrosion generally does occur. The controlled copper casting alloy B-356 has indicated Class 1 compatibility in laboratory tests, but there is no experience yet to report with the use of this material.

Some testing has been carried out on hard coat aluminum. The laboratory results of tests of 10, 20 and 30 minute penetration on aluminum alloy 6061 indicated Class 3 compatibility with H<sub>2</sub>O<sub>2</sub>. Use experience is very limited, but it may be desirable to apply hard-coat aluminum in place of 300 series stainless steel in equipment that is fabricated predominantly of aluminum. Experience is needed to determine the resistance of hard-coat aluminum to corrosion in service with intermittent wetting.

#### Stainless Steel

In general, the wrought or forged AISI 300 series stainless steel alloys with proper passivation are suitable for Class 2 service with hydrogen peroxide. Cast stainless steel is generally unsatisfactory for H<sub>2</sub>O<sub>2</sub> service unless special casting techniques are followed.

The AISI 400 series stainless steels are not suitable for H<sub>2</sub>O<sub>2</sub> service because of rusting after exposure to the H<sub>2</sub>O<sub>2</sub>. This

type of corrosion phenomenon indicates that it is necessary to examine samples from compatibility tests after the actual test has been completed. It is suggested that this examination should be made at 24 hours and one week of exposure to air after the sample is dried. It has been found that immersion of a sample in distilled water for 24 hours following the compatibility test will tend to bring out this type of corrosion of stainless steels.

Experience with use of wrought 300 series stainless steel for seamless tubing, seamless pipe, fabricated equipment for piping systems and welded tanks has been satisfactory. The use of 300 series stainless steel is recommended for high pressure flow systems and applications where presence of aluminum oxide, which can hardly be avoided in H<sub>2</sub>O<sub>2</sub> handled in aluminum, cannot be tolerated.

#### Other Metals

Many other metals have been evaluated, but few have been found suitable for H<sub>2</sub>O<sub>2</sub> service. Silicon, tantalum, tin and zirconium are exceptions. Of these, tin has been utilized to the greatest extent (for gaskets and as a solder for stainless steel).

Most other metallic elements tend toward catalytic action in contact with H<sub>2</sub>O<sub>2</sub>. This is especially true of silver, lead, cobalt, and platinum. Iron after

exposure forms iron oxide rust which is catalytic. Titanium and zinc are severely attacked by the H<sub>2</sub>O<sub>2</sub>.

Unfortunately, there are no extreme hardness metals that have shown suitability for even Class 3 service.

#### Plastics and Rubber Compounds

Since plastics and rubber compounds are organic in nature, the compatibility with hydrogen peroxide varies considerably. Even those materials which have shown excellent compatibility with 90 percent H<sub>2</sub>O<sub>2</sub> both in the laboratory and in use may be suspect when new conditions are met which have not been encountered or simulated previously. Conditions which may lead to reaction between a plastic material and H<sub>2</sub>O<sub>2</sub> are extremely varied and difficult to predict or to evaluate in the laboratory. One generalization may be observed; that is, the combination of concentrated hydrogen peroxide, organic materials and heat either from an external source or from H<sub>2</sub>O<sub>2</sub> decomposition may lead to a detonating reaction.

Higher temperature service materials such as Kel-F and Teflon have not demonstrated any indication of reaction with hydrogen peroxide over the entire concentration range. There is no known reason to avoid using those materials in any H<sub>2</sub>O<sub>2</sub> service where the H<sub>2</sub>O<sub>2</sub> will re-

main below its normal atmospheric boiling point. Mixtures of these materials with other materials must always be evaluated because reactions are varied. Glass filled Teflon is acceptable; carbon filling may or may not be; asbestos filling is not. The compatibility of the added ingredient must, naturally, be considered. Kel-F and Teflon are the most compatible plastic materials and should be utilized wherever the physical properties are suitable. It is especially recommended that these materials which have exceptionally low coefficients of friction be applied in  $H_2O_2$  service as dynamic bearings and seals without lubricants wherever possible because of the lack of compatible lubricants.

Kel-F elastomeric compounds are generally inferior to Kel-F itself in compatibility with  $H_2O_2$ . In addition, compounding materials vary and, therefore, each finished compound must be evaluated specifically before compatibility can be assured. Most of the compounds tested have met the Class 2 criteria. Experience with their use is limited so far.

Polyvinyl chloride based materials vary in their reaction with  $H_2O_2$  because of the plasticizer content and because other additives such as fillers and pigments are used. It is generally true that such additives reduce the compatibility of the compound with  $H_2O_2$ .

Koro seal 700 (molded) has been used extensively as gasketing material. The formula for this material was developed specifically for  $H_2O_2$  service in a cooperative test program with the manufacturer. Koro seal 116 and 117 (molded) are both inferior to 700 for  $H_2O_2$  service. Calendared Koro seal 117 is unsuitable because the  $H_2O_2$  penetrates into the sheet and develops gas pockets which separate the layers of material. Calendared materials which do not exhibit this condition can only be made of materials which are absolutely impermeable to  $H_2O_2$ .

Polyvinyl chloride plastics are generally permeated by 90 percent  $H_2O_2$ . This has been found to be true for both molded and plastisol types of PVC. The absorption of  $H_2O_2$  is indicated by the fact that the materials which are generally clear or translucent turn an opaque white after a period of contact with the  $H_2O_2$ . The PVC containing absorbed  $H_2O_2$  may be shock sensitive although no adverse experience of this nature has ever been experienced.

Polyvinyl chloride plastics will also leach chloride ion which in the  $H_2O_2$  will cause corrosion of aluminum even when present in minute quantities. Experience has indicated that this chloride leaching disappears on continued contact with  $H_2O_2$ , but some attack on aluminum parts can be expected before the leaching disappears. Storage first in PVC and then in aluminum is undesirable. It appears best to consider polyvinyl chloride plastics as secondary choices even for Class 2  $H_2O_2$  service.

Silicone rubber elastomers that indicate Class 2 results for  $H_2O_2$  service are considered to be superior to polyvinyl chloride materials because the possibility of chloride leaching is eliminated and

TABLE 4—Representative Results of Compatibility Tests With 90 Percent Hydrogen Peroxide—Lubricants

Material	Classification	Effect of Sample on the $H_2O_2$ , Percent Active Oxygen Loss per Week at 66°C	Impact Sensitive	Source of Material
Alkaterge C.....	4	.....	Yes	Comm. Solvents
Amino Silane Oil and Grease.....	4	Excess.	Yes	J.G. Biddle
Aplexon Hardwax W.....	4	.....	Yes	Monsanto Chem. Co.
Arochlor 1221, 1232, 1242, 1248, 1254.....	4	.....	Yes	Bardahl Corp.
Bardahl.....	4	.....	Yes	Esso Labs.
Carum 200.....	4	5.2	Yes	Carbide & Carbon
Ceresol Wax.....	4	.....	Yes	Hoover Chem. Corp.
CFE-1.....	4	.....	Yes	Hoover Chem. Corp.
Dichloro-bis-tri-fluoromethyl benzene.....	3	3.3	No <sup>1</sup>	Hoover Chem. Corp.
Dichlorohexafluorobutene.....	3	9.2	No <sup>1</sup>	Hoover Chem. Corp.
Fluorolube FS.....	2	4.2	No <sup>1</sup>	Hoover Chem. Corp.
Fluorolube FS plus 5 Percent fluorolube light grease.....	2	.....	No <sup>1</sup>	Hoover Chem. Corp.
Fluorolube heavy grease 10214.....	2	1.7	No <sup>1</sup>	Hoover Chem. Corp.
Fluorolube oil 10213.....	2	1.0	No <sup>1</sup>	Hoover Chem. Corp.
Fluorolube S.....	2	2.5	No <sup>1</sup>	Hoover Chem. Corp.
Fluorolube T.....	2	8.5	No <sup>1</sup>	Hoover Chem. Corp.
Fluorolube Oil, S-30.....	2	.....	No <sup>2</sup>	Hoover Chem. Corp.
Fluorolube Grease, Hg 1200.....	2	.....	No <sup>2</sup>	Hoover Chem. Corp.
Fluorolube Grease, GR-660.....	2	1.0	No <sup>2</sup>	Hoover Chem. Corp.
Skydrol (uncolored).....	4	.....	Yes	Monsanto Chem. Co.
Halocarbon Oil 8-25 AV.....	2	1.8	No <sup>1</sup>	Halocarbon Products
Halocarbon Grease, 25-10.....	2	.....	No <sup>2</sup>	Halocarbon Products
Halocarbon Hi-Temp Stopcock Grease.....	2	.....	No <sup>2</sup>	Halocarbon Products
Halocarbon Oil 10-21.....	2	2.9	No <sup>1</sup>	Halocarbon Products
Halocarbon Oil 11-14.....	2	2.1	No <sup>1</sup>	Halocarbon Products
Halocarbon Stop Cock Grease.....	2	1.8	No <sup>2</sup>	Halocarbon Products
Hexachlorobutadiene.....	3	3.7	No <sup>1</sup>	Hoover Chem. Corp.
Hexachloropropylene.....	4	100	.....	Hoover Chem. Corp.
Hydraulic fluid RPM.....	4	1.4	Yes	Standard Oil of California
Hydraulic Oil Houghton Safe 620.....	3	7.1	Yes	Houghton Co.
Kel-F Alkane.....	2	1.0	No <sup>2</sup>	Minn. Mining & Mfg. Co.
Kel-F Oil Cut No. 1.....	2	1.7	No <sup>1</sup>	Minn. Mining & Mfg. Co.
Kel-F Oil No. 10.....	2	3.2	No <sup>1</sup>	Minn. Mining & Mfg. Co.
Kel-F No. 90 Grease.....	2	33.2	No <sup>2</sup>	Minn. Mining & Mfg. Co.
Lindol HF (tricresyl phosphate).....	4	.....	Yes	Celanese Corp.
Lindol HFX.....	4	.....	Yes	Celanese Corp.
Liqui-Moly concentrate.....	4	.....	Yes	Climax Molybdenum
Lubrisol.....	4	.....	Yes	Arthur H. Thomas Co.
Mineral Oil.....	4	.....	Yes	Various suppliers
Paraffin.....	4	.....	Yes	Various sources
Perfluorolube grease FCD-759.....	2	10.5	No <sup>1</sup>	DuPont
Perfluorolube Oil FC-331.....	2	1.4	No <sup>1</sup>	DuPont
FC-332.....	2	1.7	No <sup>1</sup>	DuPont
FC-333.....	2	1.4	No <sup>1</sup>	DuPont
FC-334.....	2	0.8	No <sup>1</sup>	DuPont
FC-335.....	2	1.0	No <sup>1</sup>	DuPont
Petalomat.....	4	.....	Yes	Various sources
Polychloropentane (stabilized).....	4	.....	Yes	Hoover Chem. Corp.
Renex.....	4	.....	Yes	Atlas Powder Co.
Silicone XF 224.....	4	.....	Yes	Dow Corning
Silicone Oil DC-7.....	4	.....	Yes	Dow Corning
DC-44.....	4	.....	Yes	Dow Corning
DC-200.....	4	.....	Yes	Dow Corning
DC-550.....	4	.....	Yes	Dow Corning
DC-701.....	4	.....	Yes	Dow Corning
DC-702.....	4	.....	Yes	Dow Corning
DC-710.....	4	.....	Yes	Dow Corning
Silicone Oil GE 2V3733, GE 51346.....	4	.....	Yes	General Electric
Tectyl.....	4	.....	Yes	General Electric
1, 1, 2, 2, tetrafluoroethyl dodecylether.....	4	.....	Yes	.....
Tributyl Phosphate.....	4	.....	Yes	DuPont
Ucon Hydrolube U-4.....	4	8.4 (Ppt. formed)	....	Comm. Solvents Carbide & Carbon Chem.

<sup>1</sup> Non-impact sensitive to 1 kgm at room temperature.

<sup>2</sup> Non-impact sensitive to 3 kgm at room temperature.

because the flexibility of the silicones varies much less with temperature over quite a wide range. Permeability of silicone rubber to  $H_2O_2$  has not been evaluated.

#### Lubricants

The test results of possible lubricants for 90 percent  $H_2O_2$  service indicate that only the fluorinated hydrocarbons are sufficiently compatible with  $H_2O_2$  to be considered. Even these materials can probably be caused to react with the 90 percent  $H_2O_2$  if there is sufficient force

or heat applied to the mixture. However, use of fluorinated hydrocarbons has been satisfactory in transfer pump packing glands with 300 series stainless steel shafts.

Use of fluorinated hydrocarbons as lubricants for aluminum threads or in conjunction with aluminum with a high surface area in applications where heat may be created, which is a natural condition in all applications requiring lubricants, may lead to detonating reactions without any  $H_2O_2$  present.

There absence of a flame indicates that it appears hydrocarbons materials should be tested. The need for

In evaluating ice, the greatest significance is no standard producibility. The modified Tester used is simple to be generally variations in ton and weight plunger or a lubricant with negative results shall be checked.

#### Compatibility

In general reported here Becco 90 found that 90 percent  $H_2O_2$  with lower instances of 52 percent than percent  $H_2O_2$  higher compatibility tests of materials.

The results indicated that, able for 90 percent

#### TABLE 5

1060 Aluminum
3032 Aluminum
7022 Aluminum
6061 Aluminum
3024 Aluminum
355 Cast Aluminum
356 Cast Aluminum
304 Stainless Steel
316 Stainless Steel
347 Stainless Steel
Polyethylene
Kel-F
Teflon
Vinylite Vu 1
X-No classification

There also is some indication that presence of a fluorinated hydrocarbon in intimate contact with an organic material and 90 percent  $H_2O_2$  will cause the latter mixture to be of greater sensitivity than it would be alone. This phenomenon has not been adequately evaluated; therefore, it appears that the use of fluorinated hydrocarbons in conjunction with organic materials in concentrated  $H_2O_2$  service should be avoided unless the specific system to be used has been adequately tested. The best practice is to eliminate the need for lubricant if it is at all possible.

In evaluating lubricants for  $H_2O_2$  service, the results of the impact test are of greatest significance. Unfortunately, there is no standard impact tester and the reproducibility of most testers is only fair. The modified Bureau of Mines Impact Tester used for the results reported here is simple to use and has been proven to be generally reliable, but it is subject to variations in results with different operators and with the condition of the anvil plunger or weight. Therefore, although a lubricant or grease may pass all tests with negative results and then give a positive result in a later check test, it shall be classed as unsuitable for  $H_2O_2$  service.

#### *Compatibility of Materials with Becco 98 Percent $H_2O_2$*

In general, the compatibility tests reported herein have been performed with Becco 90 percent  $H_2O_2$ . It has been found that materials suitable for 90 percent  $H_2O_2$  service are suitable for use with lower concentrations and in a few instances materials unsuitable for 90 percent  $H_2O_2$  service are suitable for below 52 percent  $H_2O_2$ . However, it is not certain that all materials suitable for 90 percent  $H_2O_2$  service will be suitable for higher concentrations. Therefore, compatibility tests were run on a selected number of materials with 98 percent  $H_2O_2$ .

The results presented in Table 5 indicated that, in general, the materials suitable for 90 percent  $H_2O_2$  service are

suitable for 98 percent  $H_2O_2$  service. Some plastics are attacked more severely by the more concentrated  $H_2O_2$  due to higher energy, greater oxygen content and possibly greater solvent power. With metals, 98 percent  $H_2O_2$  generally shows less active oxygen loss than does 90 percent  $H_2O_2$ . This greater stability of highly concentrated hydrogen peroxide in the presence of metals was found to result in increased storage stability in the pure aluminum shipping drums used for all  $H_2O_2$  shipments.

#### **Design and Fabrication Techniques Recommended for $H_2O_2$ Storage and Handling Equipment**

Proper design and attention to careful fabrication by approved techniques are next in importance to the proper selection of materials of construction. Naturally, both considerations affect the selection of materials which is a compromise of these factors with the  $H_2O_2$  compatibility factor. It has been found that the time spent in careful development of tank and equipment design followed by frequent checks on the techniques utilized during fabrication of the tank or item of equipment is worthwhile. The item given this close attention and produced carefully by experienced personnel will generally be passivated most easily and give the most satisfactory service.

#### **Problem Areas Which Remain**

Although a great deal of work has been done to evaluate materials of construction for suitability for use with hydrogen peroxide solutions, entirely satisfactory recommendations are not available for all material applications. Some of the more prominent of the problem areas are listed below.

1. The corrosion of aluminum and aluminum alloys and means of its prevention need more extensive study. A better laboratory test is needed to determine the corrosion resistance of aluminum under various conditions which might be expected in hydrogen peroxide service.

Preventing corrosion of aluminum in equipment made of aluminum and other metals would allow the use of light weight aluminum parts in high pressure feed systems without introducing excessive maintenance requirements. Therefore, metallic couples with aluminum alloys need to be systematically investigated. It also would be desirable to determine whether or not couples are formed between the various aluminum alloys in  $H_2O_2$  solutions. A study of potentials developed would be an excellent guide to bi-metal system selection.

2. There is insufficient information on compatibility of metals with surface hardness above Rockwell C45. More materials need to be evaluated. Improved passivation procedures might produce improved results from some of the materials that have been tested. Evaluation of highly polished samples should be performed. Various hardening processes should be evaluated for various metals with various means of passivation. Hard chrome plate and hard anodized aluminum need to be studied more fully. There seems to be an increasing need for hard surface materials for pumps and valves for concentrated  $H_2O_2$  service. Self-lubricating materials might also be utilized in such equipment if any were chemically suitable.

3. The safety of utilizing plastic materials and lubricants for concentrated hydrogen peroxide service under conditions of elevated temperatures needs further study. A test procedure is required for evaluating the probability of the occurrence of a detonating reaction under a specific set of conditions.

4. Lubricants are required with lubricating properties and inertness to oxidation and reaction with aluminum superior to the fluorinated hydrocarbons. The selection of lubricants is a problem. The best lubricant to use with  $H_2O_2$  is no lubricant at all; however, this is not always practical. Therefore, development of more suitable lubricants is probably the most important of the problem areas suggested here.

5. For all metals there may be more satisfactory methods of passivation for  $H_2O_2$  service. Some effort should be made continually to evaluate new procedures. In general, however, the present procedures have yielded satisfactory results.

#### **Summary and Conclusions**

Tanks, equipment and systems for storing and handling hydrogen peroxide must be made of proper materials properly prepared to yield satisfactory results in service. In the selection of the proper materials, there are three factors of primary significance: (1) compatibility with the hydrogen peroxide; (2) possibility of corrosion in service; and (3) the physical properties required by the application.

Selecting materials of construction is of primary importance but proper design, fabrication and application are also important.

Naturally, this information cannot answer all questions. Some areas where information is inadequate are pointed

TABLE 5—Results of Compatibility Tests of Materials With BECCO 98 Percent  $H_2O_2$

Material	Active $O_2$ Loss, Percent/Week 66°C	Effect on Samples	Classification
1060 Aluminum (99.6).....	1.5	None	1
3052 Aluminum.....	2.6	None	1
7072 Aluminum.....	4.1	None	1
6061 Aluminum.....	5.2	None	2
3024 Aluminum.....	100	Pitted	4
355 Cast Aluminum.....	100	None	3
356 Cast Aluminum.....	3.9	None	2
304 Stainless Steel.....	12.0	Slight bronzing at higher temperature for all samples	2
316 Stainless Steel.....	53.0	Slight bronzing at higher temperature for all samples	2
347 Stainless Steel.....	30.0	Slight bronzing at higher temperature for all samples	2
Polyethylene.....	2.3	None	X
Kel-F.....	8.3	None	2
Teflon.....	2.6	None	1
Vinylite Vu 1930.....	2.7	Became opaque	2

X-No classification due to unknown probability of detonating reaction if polyethylene is heated.

out. No doubt, there are others to be encountered.

It is hoped, however, that this paper will serve as a valuable guide for hydrogen peroxide systems design and use by providing realistic and practical information about materials for hydrogen peroxide service. The use of common sense based on knowledge of this and other available data will lead to satisfactory storage, handling and use of hydrogen peroxide of all concentrations.

#### Acknowledgment

The data presented in this paper has resulted from work supported by various Bureaus of the Navy Department and the Becco Chemical Division, Food Machinery and Chemical Corporation. The authors wish to acknowledge the use of data developed under contracts with the Bureau of Ships, the Bureau of Ordnance, the Bureau of Aeronautics and the Office of Naval Research. These data included herein are thus made readily available to the Department of Defense, its contractors and suppliers.

#### DISCUSSIONS

##### Question by H. Lee Craig, Jr., Reynolds Metals Company, Richmond, Virginia:

Is it to be inferred from your statement that hard-coat anodized 6061-T6 aluminum is preferred over stainless steel in an aluminum system during passivating treatment with nitric acid or during  $H_2O_2$  storage?

##### Reply by Ralph Bloom, Jr.:

Yes, on the basis of preventing galvanic corrosion of aluminum. Experience has shown that when stainless steel parts are

used in an aluminum system, there may be sufficient formation of aluminum oxide to cause sticking of solenoid or pneumatically operated valves. This may be the case even if there is no direct aluminum to stainless steel contact. It is particularly likely to occur when a system is allowed to dry and remain out of use for a period of time.

##### Comments by M. A. Scheil, A. O. Smith Corp., Milwaukee, Wisconsin:

While the author has not mentioned it specifically in his descriptions, we have found dust to be one of the best catalysts for activating strong peroxide solutions. I noted in the author's presentation that his company favors the use of 35 percent commercial (stabilized) peroxide solution for surveillance testing of stainless steel tanks. I am much in favor of this, as an experience of my company has indicated that 35 percent unstabilized peroxide solution can have a more vigorous attack than 90 percent concentrated high purity peroxide.

There seems to be a difference of opinion between the author's company's recommendations and the Naval Air Rocket Test Station specifications on the use of 35 percent high purity peroxide for surveillance testing. I find that this is also true of the experience of the DuPont Company, and have wondered, then, why our Navy continues to specify unstabilized 35 percent peroxide for surveillance testing.

##### Reply by Ralph Bloom, Jr.:

I do not believe that ordinary dust is particularly catalytic toward concentrated hydrogen peroxide. However, dust con-

taining any iron oxides such as rust or polishing rouges will be quite catalytic. To be a hazard even dust containing iron oxide must be present in fairly heavy concentration compared to the quantity of  $H_2O_2$ . However, a tank containing even a small amount of iron oxide will probably not meet the specified criteria. In interpreting the results of passivity checks of tanks one must consider the use of the tanks. Storage tanks must be of Class 1 quality. Feed tanks generally need only to be Class 2. Sometimes, specifications are overly severe due to inexperience of the specifier; the government probably leans toward the safe side.

The problem that government establishments using 90 percent  $H_2O_2$  face when it comes to using 35 percent  $H_2O_2$  for conditioning purposes is that they have no other use for the 35 percent  $H_2O_2$  and fear a mix-up if both concentrations are stocked. Therefore, it is preferable to buy 90 percent  $H_2O_2$  and dilute it. We now accept the approach and recommend the use of diluent water which Becco has checked by means of the hot stability test, the addition of 150 mg/liter of tetrasodium pyrophosphate ( $Na_4P_2O_7$ , anhydrous) as stabilizer and adjustment of the apparent pH of the stabilized  $H_2O_2$  with reagent grade phosphoric acid to the range 2.0 to 2.4. This recommendation has been made to the U. S. Naval Air Rocket Test Station.

The use of  $H_2O_2$  for conditioning has been the subject to progressive evolution which has resulted from differences of opinion. Our recommendations have been based as much as possible on experience at our plants with procedures most adaptable to our operations.

**Any discussion of this article not published above  
will appear in the December, 1960 issue.**

# A Laboratory Evaluation of Zinc Anodes in Sea Water\*

By J. A. H. CARSON, W. L. M. PHILLIPS and J. R. WELLINGTON

## Introduction

THE EFFECT of composition on the operating characteristics of zinc galvanic anodes in sea water has been investigated by several groups (the American Zinc Institute in cooperation with the International Nickel Company at Harbor Island, the American Smelting and Refining Company, and the Chemical Department of the British Admiralty in the U.K.). In general, these investigators have reached the same major conclusions: that the presence of iron in zinc anodes is detrimental to performance and, in an otherwise pure zinc anode, the iron content must be held to a few parts per million. It was also found that the addition of certain alloying elements, notably aluminum or aluminum plus one other metal, results in a more effective anode, and permits an increase in the amount of iron which may be tolerated. Thus, these investigators were successful in pinpointing the cause of failure of commercial anodes and were able to suggest or show that the use of certain zinc alloys would result in galvanic anodes with improved characteristics.

While the results of the earlier investigators were in accord on the major factors mentioned above, the effect of more detailed factors was not so clearly defined, and even somewhat conflicting. Specifically, these points were: (1) The effect of very low levels of iron, (2) The choice and usefulness of ternary alloying additions (to a zinc-aluminum alloy), (3) The quantity of iron that may be tolerated in such alloys, and (4) The effect of current density and time

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on anode performance. In fact, it seemed highly probable that it was the lack of information on the latter two factors that tended to produce the apparently divergent results noted in the earlier work.

Teel and Anderson<sup>1</sup> found that the inactivity of high iron zinc anodes could be attributed to the electrical resistance of the films of anode dissolution products formed on the anode. It seemed reasonable to suppose that film formation and thus anode performance, might be affected by the rate of dissolution (current density) of the anode for each specific composition and by the length of time the anode films may have been allowed to accumulate. Therefore it seemed desirable to test chosen compositions at selected current densities and to measure the potential of the anode during operation. The polarized potential of the anode would be the criterion of performance, and the preferred anode composition would be the one that showed the least degree of polarization with time over a wide range of current densities. Also, this type of information would be useful for predicting or analyzing the performance of anodes in actual service.

## Test Procedure

In order to test galvanic anodes at controlled current densities, and in order to be able to measure the polarized potential of the anode during operation, it is necessary to have a test set-up with an anode-cathode geometry in which the current distribution on the anode surface is uniform and the IR drop in the electrolyte may be calculated. For these reasons an anode/cathode geometry of concentric cylindrical electrodes with shielded ends, similar to the one used

## Abstract

Previously reported investigations into the characteristics of zinc galvanic anodes in sea water have been confined to studies of the effects of composition under conditions of uncontrolled or a single controlled anode current density. Since the preferred galvanic anode alloy is one that will show little anodic polarization with time over a wide range of anode current densities, it is necessary to include current density as a factor when comparing alloy performance.

This paper presents data on the anodic polarization of various zinc alloys in natural sea water. The experimental technique and procedures employed are described. The combined effects of iron, as an impurity, over the range of 0.003 percent to 0.0320 percent, and aluminum additions over the range 0 to 22 percent, were investigated at five controlled anode current densities ranging from 0.5 to 20 ma/in<sup>2</sup>, for times up to 100 days. The interrelated effects of iron, aluminum, current density and time are presented by means of graphs and charts.

The test results confirm the major conclusions of earlier investigators with respect to the detrimental effect of iron and the beneficial effect of small aluminum additions. The results show that some of the anomalous results of the earlier work may be explained on the basis of the interrelated effects of composition, current density, and time. Within the range of compositions tested, the preferred zinc anode alloy is shown to be one containing 0.3 to 0.6 percent Al with the iron content controlled in the range 0.001 to 0.005 percent. 5.2.2

by Crennell and Wheeler<sup>2</sup> was chosen. The selected anode and cathode diameters were 4" and 18" and the rest of the equipment was so proportioned that 100 anodes could be tested simultaneously.

TABLE 1—Properties of Sea Water During Periods of Tests

	Test 1	Test 2
Temperature (°C).....	11–12	8.5–12
(°F).....	52–54	47–54
Resistivity (ohm-cm).....	29–30	29–30

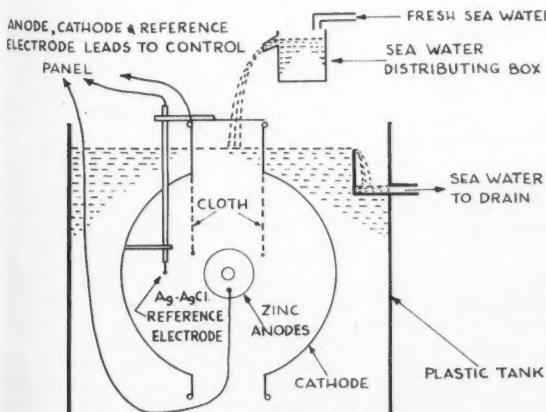


Figure 1—Cross section of anode-cathode assembly in tank.

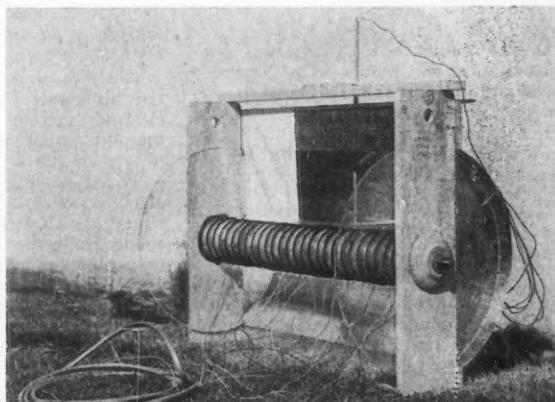


Figure 2—Anode-cathode assembly.

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Carson



Phillips



Wellington

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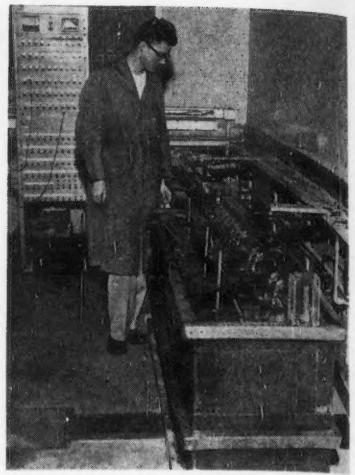


Figure 3—Test apparatus.

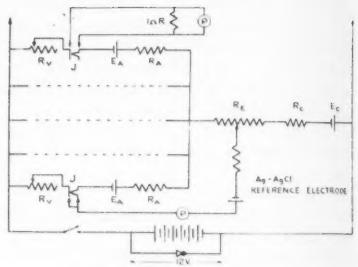


Figure 4—Electrical circuit for zinc anode tests.

TABLE 2—Levels of Independent Variables Used in Tests

CURRENT DENSITY		IRON, PERCENT	ALUMINUM, PERCENT		
			Test 1	Test 2	
ma/in <sup>2</sup>	amp/ft <sup>2</sup>			Low Range	High Range
0.5	0.07	.0003	0	0.06	1.1
1.3	0.2	.0010	0.3	0.13	2.4
3.2	0.5	.0032	1.2	0.25	5.0
8.0	1.2	.0100	..	0.5	10.5
20.0	2.9	.0320	..	1.0	22.0

(Figures 1, 2, and 3). The anodes were mounted on an insulated steel rod and were insulated from one another. All anodes operating at the same current density were mounted on one spool concentric within a common cathode. Each anode was connected by an insulated wire to the control panel. A silver/silver chloride reference electrode was positioned a known distance from the anode at a position approximately halfway between the anode and cathode. Gaps at the top and bottom of the cathode permitted the entry and exit of fresh sea water, the temperature and resistivity of which are tabulated in Table 1.

#### Electrical Circuit

Figure 4 is a schematic drawing of the electrical circuit. Each anode circuit consists of a variable resistance  $R_v$ , a zinc anode with potential  $E_A$  and film resistance  $R_A$ , the resistance of the electrolyte path  $R_E$ , the cathode with resistance  $R_C$  and potential  $E_C$ , and a source of 12 volts D.C. The current flowing through each anode was measured (with a meter

plugged into microphone jack J) and maintained at the desired value by adjusting  $R_v$ . Current densities were adjusted and anode potentials were measured with a potentiometer and the Ag/AgCl reference electrode. Measurements were made at least once a day for the first week and less frequently thereafter.

The measured potential is the potential difference between the reference electrode and the polarized potential of the anode plus the IR drop in the electrolyte to the reference electrode. The polarized potential of the anode is the algebraic sum of the anode reaction potential, the IR drop through any adherent anode films, the concentration polarization, plus any other polarization effects. Thus, the measured potential of the anode, corrected for the IR drop in the electrolyte, is the polarized or effective working potential of a specific anode, operated at a particular current density for a specified length of time. It is these polarized or effective working potentials which are so important in practice and

which form the basis of the results of this paper.

#### Test Patterns

Four independent variables were studied in this investigation: anode current density, iron content, aluminum content and time. The ranges and experimental levels selected were chosen with a view to covering the range likely to be encountered in practice and to establish optimum levels or working limits. Two series of tests were run. The first test series was designed primarily to study the effect of iron on pure zinc. Five main levels (plus two additional levels) of iron and five current densities were selected. An additional study included the effect of aluminum at two levels (see Table 2).

The second test was designed primarily to study the effect of Al in the range below 1 percent where the first test indicated the presence of an optimum level. A second smaller pattern was designed to explore the effect of Al in the range 1 percent to 22 percent. Two other small patterns were designed to evaluate a Zn-Al-Cd and a Zn-Al-Si alloy. In all of these patterns the same five levels of Fe and five levels of current density were used as in the first test series.

To keep the number of specimens

within practical limits, statistically designed patterns were employed in both test series.

All the required alloys were prepared from Special High Grade zinc with 0.0003 percent iron. Alloying additions were made with pure iron and either 1100 (99.5 percent) or high purity (99.99 percent) aluminum. Melts were made in graphite pots and specimen billets were cast in graphite molds. The analysis of each alloy was checked for Pb, Cd, Cu and Al spectrographically and for Fe by the dipyridine method. Actual analyses were very close to nominal levels.

### Results

The following series of graphs with brief explanations and conclusions will give a picture of the more important results of these tests. As four independent variables, CD (current density), Fe, Al and time, and one dependent variable, polarized potential, were studied, it is necessary to graph the results in several ways in order to show the separate and interrelated effects. Generally speaking a logarithmic scale has been used on the horizontal axis in order to cover the range in the variable represented. Also, in the graphs to follow, the more anodic potentials (more negative potentials in the sign convention used here) are plotted in the downward direction. Thus, the lower curves on any graph represent superior performance.

The effect of CD, Fe and time on pure zinc will be considered first. Figure 5 is a plot of potential versus time for anodes with four levels of Fe at the lowest, intermediate and highest CD levels. The detrimental effect of increasing iron content is evident at all CD levels. The effect of time is pronounced only at the higher Fe levels.

The effect of CD is shown in Figure 6 where potential is plotted against the CD. This is a special type of polarization curve, each point being obtained by operating one particular anode at the CD shown for the specified length of time. It can be seen that the effect of CD varies with the Fe level. At low Fe contents, the potentials are more anodic for anodes operated at low and high CD's than at intermediate CD's. At higher Fe levels, the potentials become increasingly cathodic with increasing CD. Note the change in pattern in the range 0.0010 to 0.0020 percent Fe.

In Figure 7 (potential versus percent Fe), the specific effect of iron is shown more clearly. At low CD's (0.5 to 3.2 ma/sq inch), the potential rises slowly, levels off, then rises again as the Fe content is increased. However, at the higher CD's (8 and 20 ma/sq inch) the potential rises very rapidly above about 0.0010 to 0.0020 percent Fe.

This difference in behavior at low and high CD may explain the apparently divergent results reported by Teel and Anderson<sup>1</sup> and by Reichard and Lennox.<sup>3</sup> The Teel-Anderson anodes operated at CD's in the range 10 to 20 ma/sq inch. These investigators found a sharp decrease in the current output of anodes with more than .0015 percent Fe. The commercial size anodes tested by Reichard and Lennox

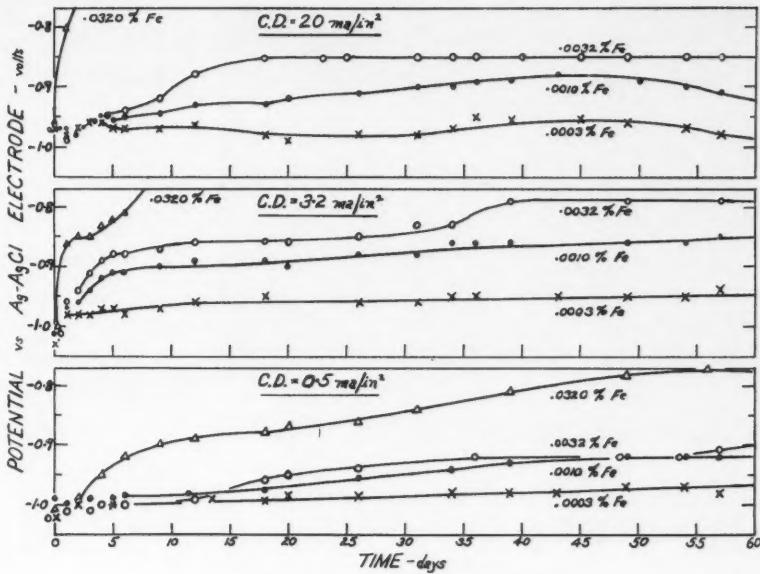


Figure 5—Potential vs time for zero percent aluminum anodes (Test 1).

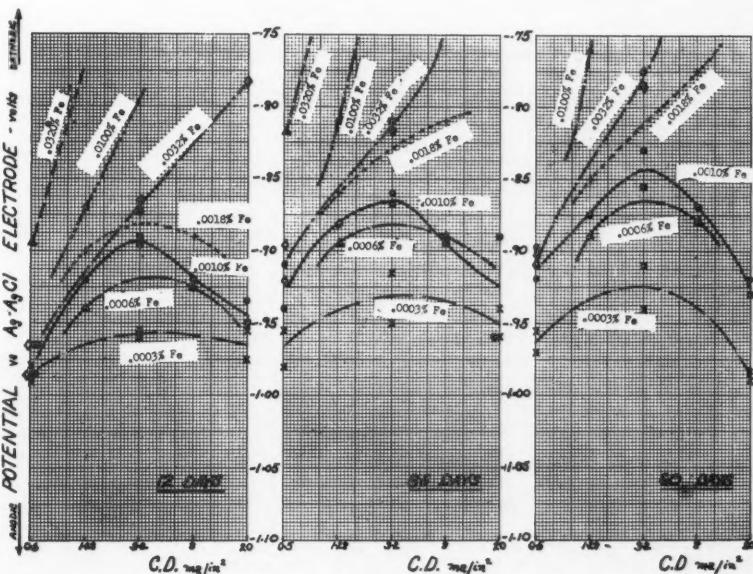


Figure 6—Potential vs current density for zero percent aluminum anodes (Test 1). Time period for the three graphs are, left to right, 12 days, 36 days, 60 days.

operated at lower CD's (approximately 3 ma/sq inch) and over an appreciably longer time. These investigators found no break in the current output curve, as the Fe content of the anode was decreased.

The combined effects of CD, Fe and time on potential are further illustrated by Figures 14(a) and 15(a) which are isometric projections of potential versus CD versus Fe at 12 and 60 days respectively. The three-dimensional surfaces formed by these variables are indicated by sections at the five CD levels, which

are the same curves shown previously in Figure 6.

On the basis of these tests the following conclusions may be made regarding the effects of Fe, CD and time on the performance of pure zinc anodes with no other additions:

1. The presence of Fe is detrimental at all levels of Fe tested, the precise effect varying with the current density. A basic change in the behavior pattern occurs in the range 0.0010 to 0.0020 percent Fe at high current densities.
2. Pure zinc anodes containing vary-

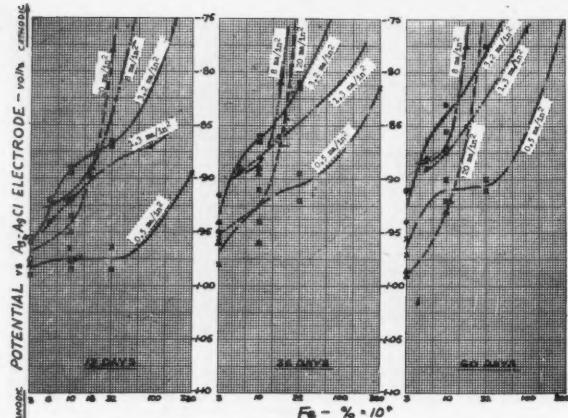


Figure 7—Potential vs iron content for zero percent aluminum anodes (Test 1). Time periods for the three graphs are, left to right, 12 days, 36 days, 60 days.

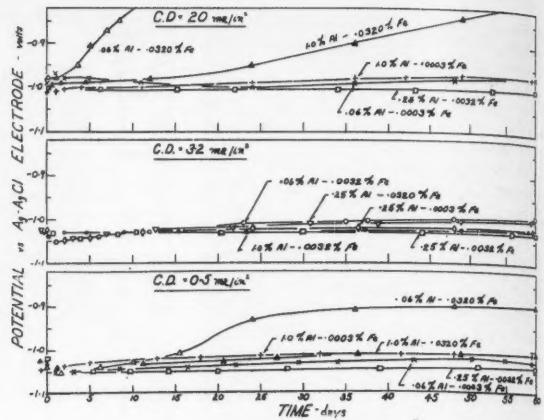


Figure 8—Potential vs time for low aluminum anodes (Test 2).

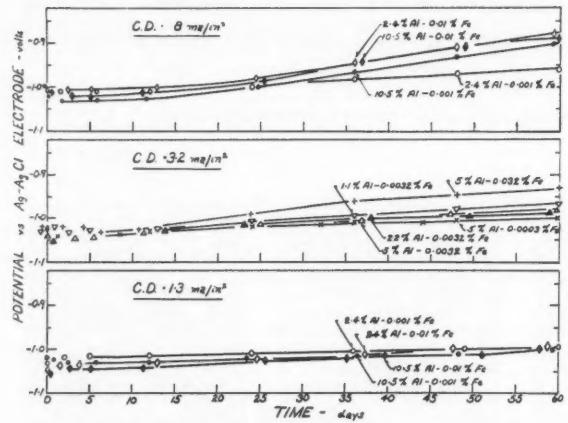


Figure 9—Potential vs time for high aluminum anodes (Test 2).

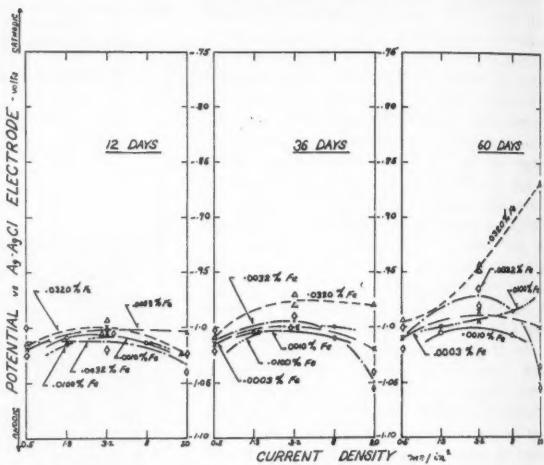


Figure 10—Potential vs current density for 0.3 percent aluminum anodes (Test 1).

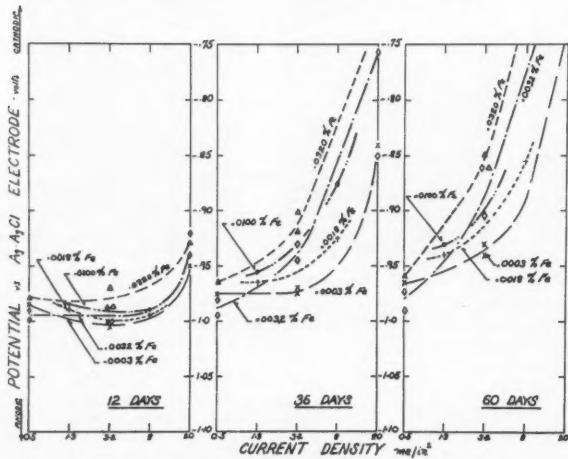


Figure 11—Potential vs current density for 1.2 percent aluminum anodes (Test 1).

ing amounts of Fe and operated at constant current density tend to polarize with time. This effect is very small for the 0.0003 percent Fe level but is significant for the 0.0010 percent level and increases markedly at higher Fe contents.

3. At the current density range most

likely to be encountered in practice, and in an otherwise pure zinc anode, there is no optimum Fe level; the lower the Fe the better.

#### Effect of Aluminum

The effect of Al in combination with

the other three independent variables, CD, Fe and time, will now be considered. In Figure 8 the potential versus time curves are shown for several combinations of Al (between .06 and 1 percent) and Fe (between .0003 and .032 percent) at the lowest, intermediate and highest

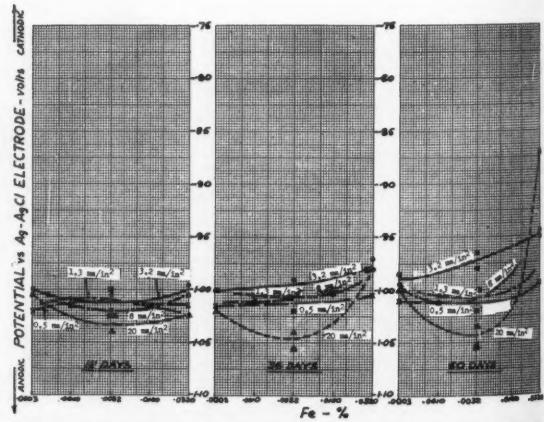


Figure 12—Potential vs iron content for 0.3 percent aluminum anodes (Test 1). Time periods for the three graphs are, left to right, 12 days, 36 days, 60 days.

CD levels illustrated on anode p for those a of Fe.

Figure time curve in the ran 0003 to middle C these high insignifi at the high anodes sh 0 percent they are a the low optimum percent Al the results

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Figure Fe versus ma/sq in plot of the results second tes analysis. N by this gr flat at the is the op optical anal current density in current density sq inch). optimum in CD bu will not c g, compo 0.25 per showed ne Note th

CD levels. The most important point illustrated here is that the effect of time on anode polarization is negligible except for those anodes containing large amounts of Fe.

Figure 9 shows the potential versus time curves for several Zn-Al-Fe alloys in the ranges 1.1 to 22 percent Al and .003 to .032 percent Fe, at the three middle CD levels. The effect of time at these higher Al levels is small but not insignificant at low CD's, and appreciable at the higher CD's. While the high Al anodes show less polarization than the 0 percent Al anodes at high Fe levels, they are appreciably more polarized than the low Al anodes. Thus, there is an optimum Al addition in the range 0-1 percent Al. This result is in accord with the results of other investigators.

In the low Al range, Figures 10 and 11 show the relationship between potential and CD at the 0.3 and 1.2 percent Al levels respectively. At 0.3 percent Al, CD has only a minor effect on potential at 12 days, at all Fe levels tested. At 60 days the highest Fe anodes show marked polarization, but the lower Fe anodes are relatively unchanged. At the 1.2 percent Al level the behavior of all Fe containing anodes is similar and the potentials tend to become more cathodic with increasing CD. This effect increases markedly with time. That is, the effect of CD on anode polarization is less at the 0.3 percent Al level than at either the 1.2 or 0 percent Al levels (see Figure 6).

Figures 12 and 13 illustrate the same results plotted as potential versus Fe curves. These three graphs show very clearly that the 0.3 percent Al alloy will tolerate much more Fe than either the 0 or 1.2 percent Al alloys.

The isometric projections in Figures 14 and 15 show the interrelated effects of all four variables on anode potential. The surface for the 0.3 percent Al alloy at 12 days is virtually horizontal with little curvature, again showing the very minor effects of Fe and CD on the polarization of this alloy. At 60 days the high CD-high Fe corner (for this alloy) has been moved up somewhat, illustrating the effect of time on these variables at the higher levels. The surface for the 1.2 percent Al alloy is markedly affected by time.

Figure 16 is a plot of potential versus Fe versus Al at 60 days and the 3.2 ma/inch CD level. This graph is a plot of the quadratic equation fitted to the results in the low Al pattern in the second test series by means of statistical analysis. Note that the surface indicated by this graph is bowl shaped, relatively flat at the bottom, at the center of which is the optimum composition. The statistical analysis shows the optimum composition is the same over the range of current densities tested (0.5 to 20 ma/inch). The potential assigned to the optimum will vary slightly with changes in CD but the location of the optimum will not change. As illustrated by Figure 8, compositions close to the optimum (0.25 percent Al - .0032 percent Fe) showed negligible polarization with time. Note that the surface enclosed within

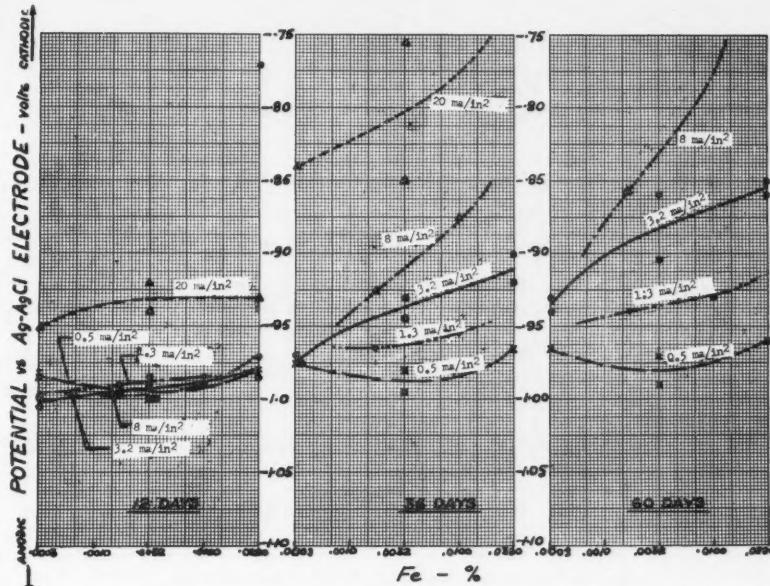


Figure 13—Potential vs iron content for 1.2 percent aluminum anodes (Test 1). Time periods for the three graphs are, left to right, 12 days, 36 days, 60 days.

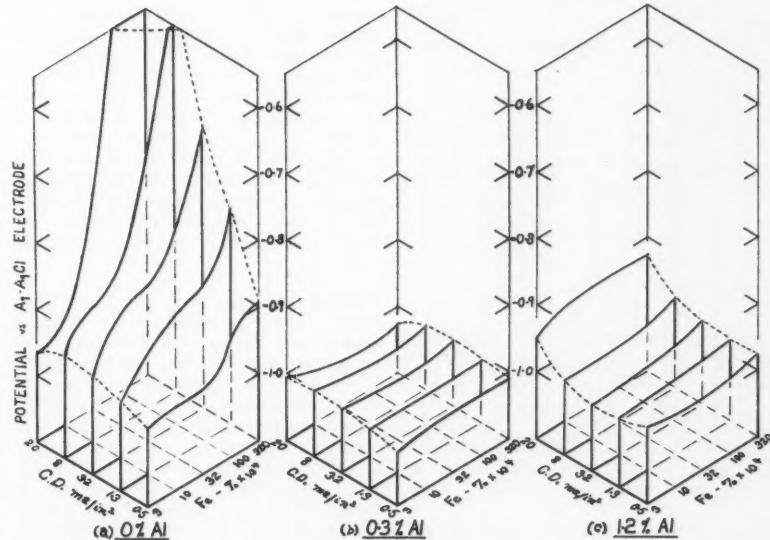


Figure 14—Potential vs current density vs iron content at 12 days.

the —1.03 volt isopotential is relatively flat so that for practical purposes the preferred composition may be defined as a range of compositions within fairly broad limits. The rectangle enclosed by the dotted lines defines a suggested optimum composition range with limits of 0.3 to 0.6 percent Al and 0.0010 to 0.0050 percent Fe.

To sum up, on the basis of the foregoing tests, the following conclusions may be noted:

1. The presence of Al in zinc results in an anode with markedly improved performance and greatly increased tolerance for Fe.

2. Over the range of compositions tested, 0 to 22 percent Al, there is an optimum Al level in the range 0.3 to 0.6 percent Al. Larger or smaller additions are less effective.

3. For an anode with the optimum Al content, there is an optimum Fe content.

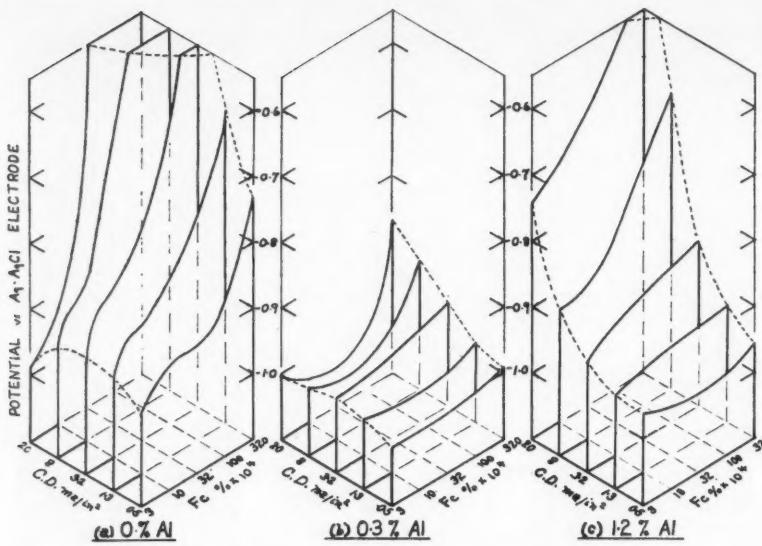
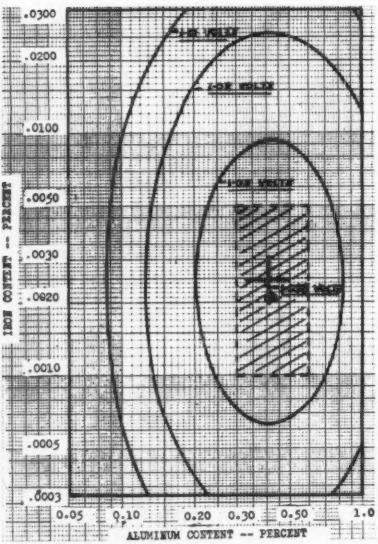


Figure 15—Potential vs current density vs iron content at 60 days.

Figure 16—Anode potential vs iron and aluminum contents. Low aluminum pattern (Test 2). Time was 60 days; current density was 3.2 ma/sq. inch. Cross is at  $-0.035$  volt; rings going outward are  $-0.03$ ,  $-0.02$  and  $-0.01$  volts, respectively.

The iron should be less than 0.0050 percent and more than 0.0010 percent.

### Third Alloying Element

Other investigators have reported that the addition of a third alloying element to a zinc-aluminum alloy produced an anode with superior properties. The polarization characteristics of two such alloys have been studied in these tests. In these tests the effect of CD, Fe and time on the polarization of only two specific alloys was studied, with no attempt made to determine the optimum compositions of the ternary alloys.

Figure 17 shows the potential versus time curves for a 0.25 percent Al, a 0.5 percent Al-0.07 percent Si and a 0.1 percent Al-0.05 percent Cd zinc alloy, each containing 0.0032 percent Fe, at the lowest, intermediate and highest CD levels. At 0.5 and 3.2 ma/sq inch, time has a negligible effect on the potential of these alloys and, in fact, there is no real difference between the three alloys. At 20 ma/sq inch the Zn-Al-Cd alloy polarizes significantly and this effect increases with time. All three alloys are slightly less anodic at the highest CD level.

When the tests at constant current density were terminated, the current density was changed to different levels for periods of a few days and the change in anode potential observed. The results of one such current variation test are plotted in Figure 18 for the Al-Si and Al-Cd ternary alloys and for a number of aluminum containing alloys. In this test the current density for a group of anodes which had previously operated at 3.2 ma/sq inch for 100 days was changed from 3.2 to 20 to 0.5 and back to 3.2 ma/sq inch. Thus the curves in Figure 18 are normal polarization curves as they represent the change in polarized potential of an anode operated over the range of current densities shown.

All anodes polarized with increasing current density but to varying degrees. This polarization in most cases was reversible. The alloy which will be best able to meet the demand for increased current output is the one with the lowest and flattest polarization curve. On this basis the 0.5 percent Al-0.07 percent Si alloy performed better than the other alloys tested, except perhaps at the 0.032 percent Fe level. However, the reproducibility of the data at the 20 ma/sq inch

current density level was not too good (as shown by the duplicate samples of the Al containing alloys). Also at the 0.0003 and 0.0032 percent Fe levels and at current densities below 10 ma/sq inch the behavior of the Al-Si, 0.25 percent Al and Al-Cd alloys was similar with some slight superiority in the order noted.

That there is an optimum Al and Fe addition is again illustrated—note that at the 0.0032 percent Fe level the 0.25 percent Al alloy is much better than the 0.06, 1.0 and 5.0 percent Al alloys and that the 0.25 percent Al-0.0032 percent Fe alloy is somewhat better than the 0.25 percent Al-0.0003 percent Fe alloy. It is also interesting to note that, at the 0.0032 percent Fe level, the curves for the 0.1 percent Al-0.05 percent Cd and 0.5 percent Al-0.07 percent Si alloys occupy the positions one might anticipate being held by the simple 0.1 percent Al and 0.5 percent Al alloys (see Figure 16). Unfortunately, in the statistical layout of the tests, the 0.1 and 0.5 percent Al alloys were not included in the groups of anodes subjected to current variation tests and for that reason a direct comparison cannot be made. The results suggest though that the effect on anode polarization of the ternary alloying addition tested is small.

The conclusions to be drawn from this series of tests are:

- For anodes operating at constant current density a zinc alloy containing only Al in the optimum amount performs as well as the two ternary alloys tested (Zn-0.1 percent Al-0.05 percent Cd and Zn-0.5 percent Al-0.07 percent Si).

- The Zn-0.25 percent Al, Zn-0.5 percent Al-0.07 percent Si and Zn-0.1 percent Al-0.05 percent Cd alloys with low to intermediate iron contents, respond similarly to changes in current demand over the normal operating range, 1 to 8 ma/sq inch.

- The current variation tests further emphasize the effect of iron and aluminum content on the performance of zinc anodes. Alloys within the optimum compositional range will show markedly superior response to changes in current demand.

In summary it may be said that:

- The test results confirm that the effect of composition, time and current density on anode performance are interrelated and that any test that has as its objective the definition of an optimum anode composition must take into account all three factors.

- The test results confirm the work of the earlier investigators with respect to the effect of major changes in composition, and, on the basis of a more complete understanding of the interrelated effects of composition, CD and time, permit an explanation of some of the previously observed anomalies.

- A preferred zinc anode composition for use in natural sea water has been defined.

In conclusion it may be said that further studies on the effect of water jets on the adherence of anode films have



been carried out to determine characteristics of the planned structure. The moderate characteristics that a better alloying element may be obtained.

Permit this investigation

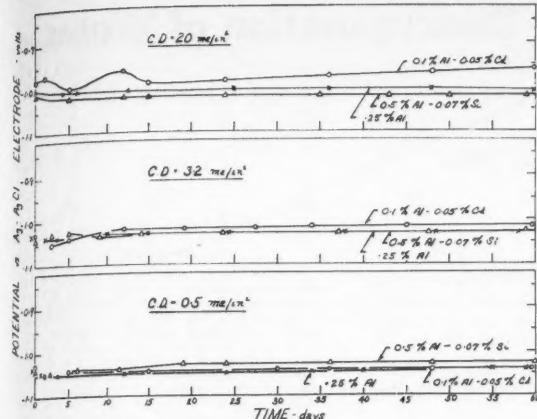


Figure 17—Potential vs time for preferred compositions with 0.0032 percent Fe (Test 2).

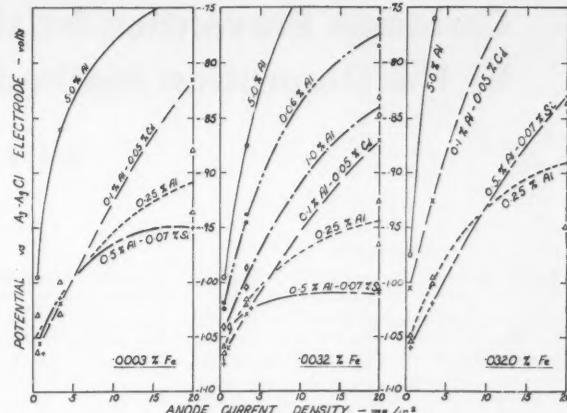


Figure 18—Anodic polarization with current density changes after operating 100 days at 3.2 mA/sq inch (Test 2).

been carried out and that further work to determine the nature and characteristics of the anode dissolution products is planned. A study of the metallographic structure of the alloys and its relation to the mode of anode dissolution and other characteristics is also planned in order that a better understanding of the effect of alloying additions on the performance characteristics of zinc galvanic anodes may be obtained.

#### Acknowledgments

Permission to describe the results of this investigation was granted by the

Defense Research Board of Canada and The Consolidated Mining and Smelting Company of Canada Limited.

The authors express their thanks to D. J. Evans, Pacific Naval Laboratory, for his advice on the electrical circuitry, Mr. H. L. Williams, The Consolidated Mining and Smelting Company of Canada Limited, for the statistical analyses and designs, Messrs. G. Brown and C. J. Mitchell, The Consolidated Mining and Smelting Company of Canada Limited, for their work on the chemical and spectrographic analyses, and Mr. R. Buckett, Pacific Naval Laboratory, for

his assistance in setting up and operating the test. The experimental equipment was provided by Cominco and the experimental facilities by the PNL Corrosion Laboratory.

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**Any discussion of this article not published above will appear in the December, 1960 issue.**

# Corrosion Prevention by the Deoxygenation of Water By the Desorption Method\*

By P. A. AKOL'ZIN

## Introduction

THE ESSENCE of the desorption method for the deoxygenation of water, which was proposed by P. A. Akol'zin and V. V. Glushenko,<sup>1</sup> consists in the following: If in a vessel there is a volume  $v_1$  of water containing  $c$  mg/liter of oxygen and a volume  $v_2$  of gas not containing oxygen, the system is initially not in equilibrium in relation to the distribution of oxygen between the liquid and the gas phases. Therefore the oxygen contained in the water begins to diffuse into the gas phase. The diffusion process will continue until the equilibrium state  $O_2 \rightleftharpoons O_2$  is established, whereby the oxygen is distributed between the water and gas in accordance with Henry's Law:

$$\frac{c_k}{p} = k_1 \quad (2.11)$$

where  $c_k$  = residual oxygen content of the water, mg/liter

$p$  = partial pressure of oxygen in the gas, mm Hg

$k_1$  = constant (Henry's constant), the different values of which for the most practically important temperatures are shown in Table 2.5.

The desorption of oxygen from water in this way under known conditions has been found to be extremely effective, and water deoxygenated thus may be used as feed for boilers and for other thermo-technical purposes.

The amount of oxygen remaining in the water after the completion of the desorption process can be determined by calculation. The following relation exists

\* Submitted for publication March 2, 1959. This paper was extracted from P. A. Akol'zin's work entitled Corrosion of Boiler Metal (Gosenergoizdat, Moscow, 1957). The English translation printed here was prepared by J. Jackson, Chemical Engineering Services, Imperial Chemical Industries, Ltd., London, England.

between the partial pressure of oxygen  $p$  and its concentration in the gas phase:

$$p = k_2 c_2 \quad (2.12)$$

where  $c_2$  = concentration of oxygen in gas phase, mg/liter

$$k_2 = \frac{769 v}{32,000}$$

In the last equation  $v$  = the volume in liters of a gram-mole of oxygen at 760 mm Hg and at various temperatures. From Equations (2.11) and (2.12):

$$\frac{c_k}{c_2} = k_1 k_2 = k \quad (2.13)$$

The values of  $v_1$ ,  $k_2$  and  $k$  are shown in Table 2.5.

On the basis of a material balance for the oxygen in the system, its content in the water is given by:

$$c = \frac{c_k v_1 + c_2 v_2}{v_1} \quad (2.14)$$

After substituting the value of  $c_2$  from Equation (2.13), the following expression is obtained for the calculation of the final oxygen content in the deoxygenated water ( $c_k$ ):

$$c_k = \frac{c}{1 + \frac{1}{k} \frac{v_2}{v_1}} \quad (2.15)$$

The results of the calculation of  $c_k$  from Equation (2.15) are shown in Figures 2.15-2.17, together with the experimentally determined values of  $c_k$ ,  $v_2$ ,  $v_1$  and  $t$ ; the curves show the good agree-

ment between the values of  $c_k$  obtained by different methods. For  $v_2/v_1 > 5$ , even when the water is not heated, the method described above enables the oxygen content to be reduced to 0.03 mg/liter—a value which is quite acceptable as regards corrosion prevention. The effect of deoxygenation by desorption, in essence, does not depend on the nature of the gas with which the liquid is mixed.

In the practical realization of this method, the water undergoing deoxy-

## Abstract

Corrosion of boilers can be prevented by the deoxygenation of boiler water by desorption. When this method is used the water undergoing deoxygenation is mixed with a gas which contains no oxygen. Because of the diffusion into the gas of the oxygen dissolved in the water, the deoxygenation of the water takes place to the degree needed. After the conclusion of the diffusion process, the gas and oxygen are automatically removed from the water, regenerated and again returned to the system. The feeding of the gas and its intensive mixing with the water is accomplished in an ejector. The separation of the gas/water mixture into gas and water (already deoxygenated) is done by a desorber device; the regeneration of the gas is included in the reactor. Data reported include relation between degree of oxygenation and: (1) water temperature, (2) ratio of volumes of water and gas, (3) initial oxygen content, (4) water pressure at ejector, (5) water level in desorber, (6) temperature of gases used for heating reactor (for wood charcoal, coke, anthracite, and iron filings), and (7) time of contact of water and gas in mixer.

Figure 2.17—

ation is no oxygen in the gas water, the takes place the conclusion the gas are removed again ret

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The v the gas compon from the it in neg transfer the water of the g ton, wh water, a through cylindri tank of pump. oxygen, consists filled wi The hea electrical. The che separate mation as a res the cha temperature lower to  $CO_2$ . T pass

TABLE 2.5—Values of Constant  $k$  as a Function of Temperature

Temperature of Water, Degrees C	Value of $k_1$	Volume of Gram-mole of Oxygen $v$ (liters)	Value of $k_2$	Value of $k = k_1 k_2$
10	0.071	23.2	0.55	0.039
20	0.058	24.0	0.57	0.033
30	0.050	24.9	0.59	0.030
40	0.045	25.6	0.61	0.027
50	0.040	26.5	0.63	0.025

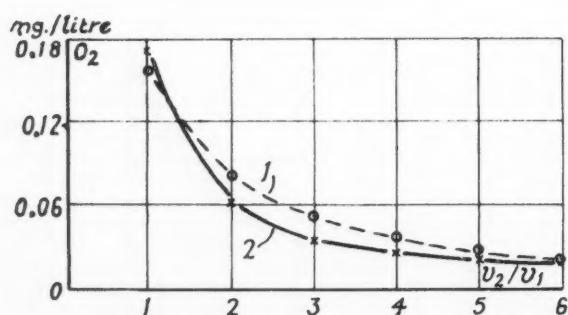


Figure 2.15—Relation between degree of deoxygenation and ratio of volumes of water and gas (for  $c = 6$  mg/liter,  $t = 40^\circ\text{C}$ ). Curve 1 is theoretical and curve 2 is experimental.

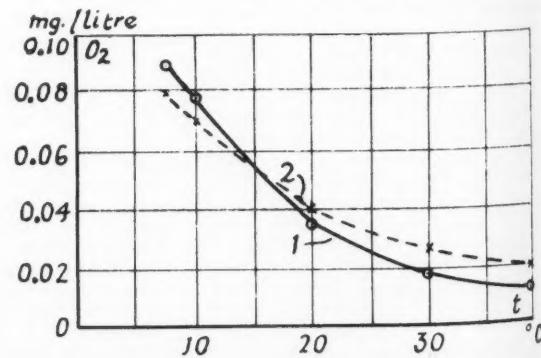


Figure 2.16—Relation between degree of deoxygenation and water temperature. Curve 1 is theoretical and curve 2 is experimental.

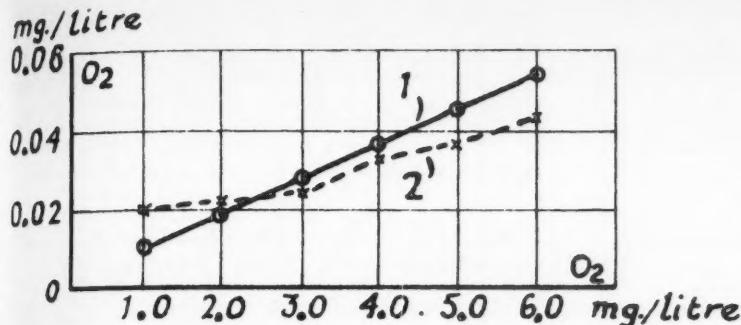


Figure 2.17—Relation between degree of deoxygenation and initial oxygen content ( $t = 40^\circ\text{C}$ ,  $V_1/V_2 = 3$ ). Curve 1 is theoretical and curve 2 is experimental.

ation is mixed with a gas which contains no oxygen. Thanks to the diffusion into the gas of the oxygen dissolved in the water, the deoxygenation of the water takes place to the degree needed. After the conclusion of the diffusion process, the gas and the oxygen are automatically removed from the water, regenerated and again returned to the system.

The feeding of the gas and its intensive mixing with the water is accomplished in an ejector. The separation of the gas/water mixture into gas and water (already deoxygenated) is effected in a special device—the desorber; the regeneration of the gas is achieved by the reactor.

A diagram giving the essentials of the simplest form of deoxygenation by desorption is shown in Figure 2.18.

The water undergoing deoxygenation is fed at a pressure of 3-4 atmospheres into the jet ejector (gas/water ejector), which maintains a continuous gas circulation in the closed system. The deoxygenation process takes place essentially in the ejector itself as a result of the intensive mixing of the gas and water, with the formation of an emulsion, which takes place in it. The process is completed in the desorber.

The water coming into contact with the gas is enriched in certain gaseous components which were either absent from the inlet water or were present in it in negligible quantities. The diffusional transfer leading to the deoxygenation of the water takes place during the passage of the gas/water mixture to the separator, where the gas is separated from the water, and the deoxygenated water is fed through the annular space formed by the cylindrical parts of the desorber into the tank of deoxygenated water or to the pump. The separated gas, enriched in oxygen, is passed to the reactor which consists of a hermetically sealed furnace filled with wood charcoal or iron filings. The heating of this material is effected electrically or by means of flue gases. The chemical combination of the oxygen separated from the water with the formation of carbon monoxide takes place as a result of the contact of the gas with the charcoal, which is heated up to a temperature of  $800^\circ\text{C}$  and above. At lower temperatures the main product is  $\text{CO}_2$ . The deoxygenated gas is then passed again to the ejector.

Thus the only material used up in the removal of the oxygen is charcoal, which is fed to the reactor in sufficient quantities to ensure continuous deoxygenation of the water for any given period of time up to several months.

Thanks to the small surface area of the reactor, the heat losses to the surrounding medium are negligible. The heat given to the water by the gas is fully utilized, since the water is to be used for thermotechnical purposes. As a result of the contact with the hot gases, the water in the desorber is heated through approximately  $0.5^\circ\text{C}$ .

Deoxygenation takes place just as effectively in a reactor filled with iron filings.

It should be noted that the deoxygenating gas entering the ejector is essentially the atmospheric nitrogen. The atmospheric air filling the system is removed in the first few minutes of operation. This takes place as a result of the oxidation of the charcoal or iron filings in the reactor. It is not necessary, therefore, to fill the system specially with gas either before starting up or during operation. Thanks to the fact that the system is closed, the supply of the solid reducing agent takes place automatically in the quantities required to combine with the oxygen separated from the water.

Experimental results have shown that the effectiveness of deoxygenation depends only slightly on the load on the apparatus and the output for which it has been designed. Thus, for example, equipment of this type is operating successfully in industry at the present time with outputs of 1-100 ton/hr and above.

The relation between the effectiveness of deoxygenation and the water pressure at the ejectors, as determined experimentally, is shown in Figure 2.19. A marked loss of effectiveness of deoxygenation occurs if the water pressure is reduced to about 1.5-2 atmospheres. At pressures above 3 atmospheres it is possible with safety to achieve a reduction of the oxygen content to 0.03 mg/liter (customary for boilers), even if the ejectors are fed with fairly cold water ( $t = 25^\circ\text{C}$ ). The improvement in the effectiveness of deoxygenation on increasing the pressure results from the increase in the ratio ( $v_2/v_1$ ), i.e., in essence, as a

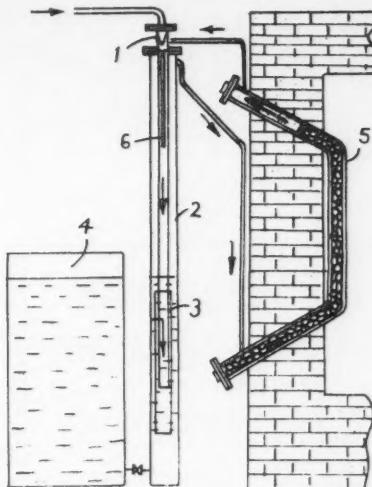


Figure 2.18—Diagram of desorption installation. 1—Ejector; 2—Desorber; 3—Separator; 4—Tank for deoxygenated water; 5—Reactor; 6—Mixer.

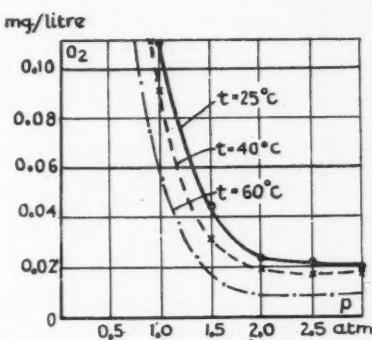


Figure 2.19—Experimental relation between degree of deoxygenation and water pressure at the ejector.

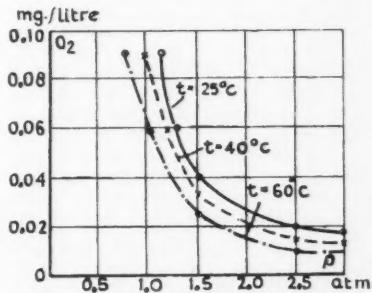
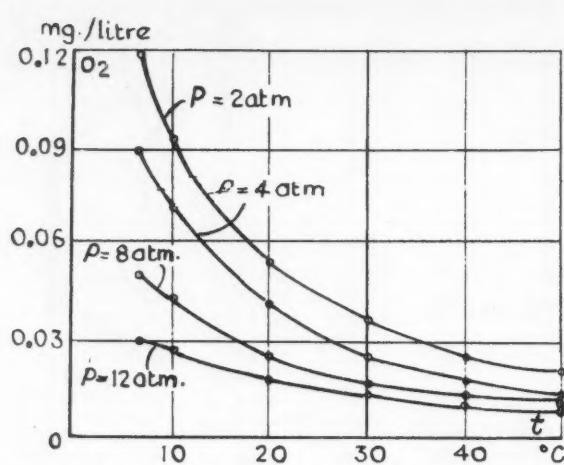


Figure 2.20—Theoretical relation between degree of deoxygenation and water pressure at the ejector.

result of the increased gas flow in the ejector. The theoretical relation between the degree of deoxygenation and the water pressure at the ejector was obtained from Equation (2.15). As Figure 2.20 shows, the calculated results are in good agreement with the experimental data.

The increase in the effectiveness of deoxygenation with increasing water temperature at constant pressure must be attributed to the increase in the intensity of the diffusion process underlying the



**Figure 2.21—Relation between degree of deoxygenation and water temperature for various pressures.**

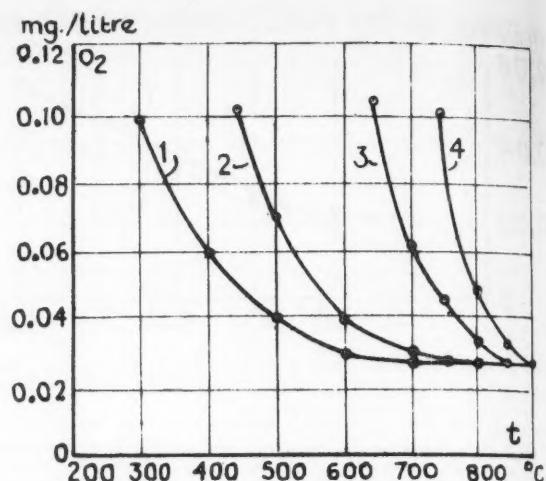


Figure 2.22—Relation between degree of deoxygenation and temperature of gases used for heating reactor. Curves are as follows: 1—wood charcoal; 2—Coke; 3—anthracite; 4—iron filings.

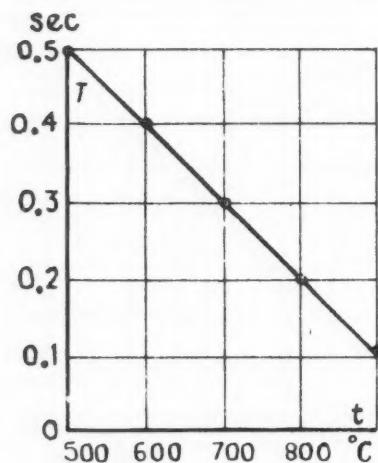


Figure 2.23—Relation between temperature and optimum residence time of gases in reactor.

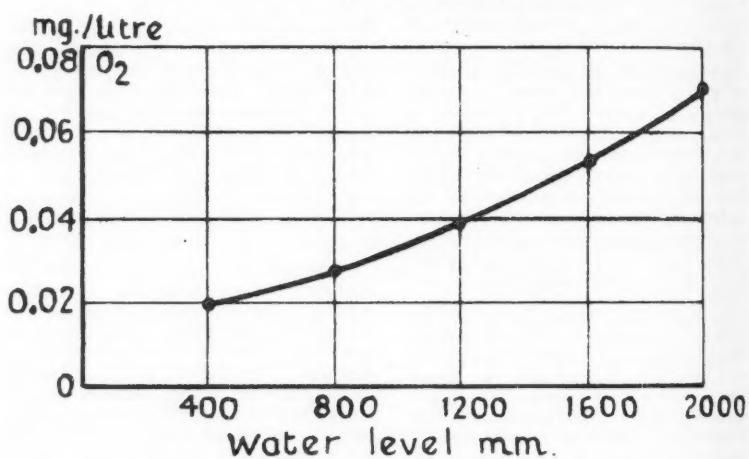


Figure 2.24—Relation between degree of deoxygenation and water level in desorber (for  $t_{\text{water}} = 40^\circ \text{C}$ ).

phenomenon of desorption. It follows from Figure 2.21, in which the relationship just mentioned is shown for various pressures, that very effective deoxygenation can be obtained even at very low water temperatures (5-10°C); for this, however, a water pressure of 10-12 atmospheres at the ejectors is required.

The choice of temperature conditions for the operation of the reactor is of great practical importance in achieving complete deoxygenation of the gas entering the reactor. Experiments on this aspect of the process were carried out with the reactor filled with wood charcoal, coke, anthracite and iron filings. The experimental results, shown in Figure 2.22, show the extremely high reactivity of wood charcoal with respect to oxygen. This reducing agent gives practically complete deoxygenation of the gas entering the reactor even at temperatures of 500-600 C. To obtain a similar effect with iron filings, it is necessary to heat to approximately 950 C; with anthracite, to 900 C; and with coke, to 600 C.

The effectiveness of operation of the reactor in absorbing oxygen is determined by the residence time in it of the gases entering from the desorber. This relationship is shown in Figure 2.23.

Data on the effect of the water level in the desorber on the degree of deoxygenation are shown in Figure 2.24. The effect is quite appreciable; the oxygen content of the water leaving the apparatus is somewhat greater for the maximum water level in the desorber.

Observations have also established that with increasing level in the absorber, the quantity of gas entering the ejector is decreased; this is obviously the result of the development of additional resistance in the column of liquid in the desorber. This is also the cause of the reduction in the effectiveness of deoxygenation of the water with increased level in the desorber. The effectiveness of deoxygenation, as shown by Figure 2.25, is directly related to the time of contact of water and gas.

## Laboratory and pilot-plant studies of

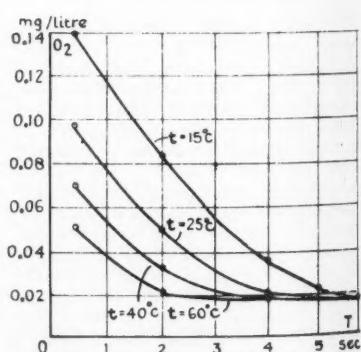


Figure 2.25—Relation between degree of deoxygenation and time of contact of water and gas in mixer.

the new method of deoxygenating water have provided initial data for the design of industrial installations of this type.

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1. P. A. Akol'zin and V. V. Glushenko. Operation of Equipment for the Desorption Deoxygenation of Water by the VTI Method. VTI Information Letter, No. 1954-6. 1954.



# NATIONAL ASSOCIATION of CORROSION ENGINEERS



## The Analysis and Composition Of Aluminum Corrosion Products\*

A Report of NACE Technical Unit  
Committee T-3B\* on Corrosion Products

**NACE TECHNICAL  
COMMITTEE REPORT**  
**Publication 60-5**

### Introduction

A CORROSION laboratory is frequently presented with a corroded piece of metal for which the cause of corrosion is to be determined. Corrosion products may be in place on the metal or may have been scraped off by the sampler and submitted separately. Usually the amount of such products is small, while the story accompanying the specimen is frequently fragmentary or nonexistent. Often there is no opportunity to obtain further information.

There are only two published general papers available on aluminum corrosion products.<sup>1,2</sup> Corrosion "detective" work in this field to date is largely a matter of trial and error. Some progress has been made, and enough experience accumulated to develop a routine chemical analytical method which is described in this report. It is recorded in an effort to develop a systematic approach and to stimulate thought on the subject.

The general corrosion behavior of aluminum has been discussed by Godard.<sup>3</sup> Aluminum is recognized to possess good corrosion resistance in many environments, and to be unsuitable for use in others. Occasionally corrosion difficulties are encountered in the former class due to poor design or installation or to the unsuspected presence of a deleterious impurity in the medium.

The greatest number of cases submitted to this laboratory have involved aluminum exposed to the atmosphere or in contact with various waters, although there have been a number of other special cases which will be described in this report.

The most common corrosion product produced in the corrosion of aluminum alloys is primarily aluminum oxide (in various states of hydration, if water or moisture is present in the environment) containing decomposition products from the alloying elements, and some constituents from the surrounding medium.

Frequently some metallic aluminum is included in the product. Analysis of the corrosion product often indicates the presence of an abnormally high amount of some constituent from the environment which may be the primary cause of corrosion. In special cases other corrosion products such as aluminum sulfate, chloride and acetate have been detected.

Unlike the corrosion products of other metals, for example iron, those of aluminum are usually amorphous to X-rays. Thus, X-ray diffraction techniques have provided little information on the subject. Occasionally, calcium carbonate (calcite), alumina monohydrate (boehmite) and aluminum silicate (mullite) have been detected by X-ray diffraction in aged corrosion products from aluminum.

Physical and chemical techniques used in determining the cause of corrosion are presented in this report in addition to data obtained on the many products investigated to date in this laboratory.

### General Procedures

*Tests with Corrosion Product in Place*

**Visual Examination of the Corroded Surface.** The physical form of the corrosion product may give an important clue to the cause of corrosion. For example, the fibrous structure of corrosion products formed as a result of mercury contamination of an aluminum surface is unique<sup>4</sup> and can be recognized with rather limited experience. In a recent case, one glance at perforated piping in a new chemical plant was sufficient to diagnose the problem as due to mercury, and this was confirmed by subsequent analysis of the corrosion product.

The type of surface attack sometimes provides a clue. Sometimes, for example, pits due to mercury, and in some instances fairly large areas, are eaten away leaving cavities which have smooth rounded contours and flat bottoms. Other examples suggest that corrosion due to mercury is not recognizable as especially different in form.<sup>5</sup>

In another case, an aircraft alcohol

### Abstract

The various techniques applicable to investigation of aluminum corrosion products are discussed. Analytical procedures suitable for the determination of the various constituents in aluminum products are outlined. Methods reported include X-ray diffraction, X-ray fluorescence, differential thermal analysis, electrographic methods, spectrographic analysis, microscopic examination, and qualitative and quantitative tests. Analytical data are presented for a number of corrosion products investigated. A study is made of corrosion products found in the following environments: fresh water, sea water and the atmosphere. Other topics discussed include presence of copper and mercury in aluminum corrosion products, pH of surface and water extract, and effect of chlorides. Data reported include effect of sea velocity on composition of corrosion products formed in 99.5 percent aluminum tubing, constituent/alumina ratios for sea water pipe products and composition of corrosion products of different alloys exposed in various industrial atmospheres.

3.4.3

tank was found to have perfectly circular pits with rounded bottoms. The absence of corrosion product and the shape of the pits suggested dissolution by drops of an aggressive chemical. Dilute nitric acid used to remove residual welding flux and which was incompletely removed by rinsing was suspected. Traces of nitrate ion were detected by a qualitative spot test in the pits.

In severe environments Al-Cu alloys suffer a lamellar form of corrosion with exfoliation or "soda cracker" at the edges. A knife blade can be inserted between the layers, sometimes to an appreciable depth. This can give the first clue to alloy identification which is usually unknown.

With experience the appearance of an aluminum surface that has been exposed to an atmosphere containing aggressive chemicals (such as hydrochloric acid or sodium hydroxide) can often be recognized from the pattern and nature of the pitting that results. Pits are random on the surface, shallow, and of irregular shape with wisps or tails radiating irregularly from a point. There are no adjacent stains. On the other hand, pits caused by condensed droplets of water are small, shallow and round (pinpoint) and are usually surrounded a short distance away by a ring or halo of dark stain.

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**Determination of Surface pH.** Before removing the corrosion products from an aluminum surface, and particularly when the amount of product available is small, it is desirable to determine the presence or absence of acidity or alkalinity on the surface. For this purpose a strip of pH-indicating test paper is placed on the surface and wetted with a small drop of distilled water. The resultant color of the wetted paper is then compared with a color chart which gives the pH of the drop extract. Others have found a one drop glass electrode preferable for this determination.<sup>5</sup>

The presence of acidity or alkalinity when matched to the conditions of exposure may give an important clue to the cause of corrosion. In one case<sup>6</sup> the surface of a piece of corroded aluminum conduit removed from a concrete floor slab (an alkaline environment) showed acidity which suggested that the surface had functioned as an active anode. This was traced to galvanic corrosion caused by contact with reinforcing steel. The concrete was found to contain chloride which provided the necessary conductivity for galvanic corrosion to occur. In another case, etched corrosion patches on the interior of tubing extruded through a porthole die was blamed on water entering the tubing during the quenching process (a quench-at-the-die water box) through poor weld fissures in the tubing. However, a pH check of the interior surface (carried out as an afterthought) revealed an acid condition (pH 3.5) which could not have come from this cause and pointed toward previously unsuspected chemical contamination.

If there is a preferred stain pattern on the surface, pH measurements should be made at different locations and may indicate a correlation between stain and surface contamination. For example, longitudinal black stains on extrusions are sometimes caused by streaks of residual alkali from a die cleaned with caustic and inadequately rinsed. It should be pointed out that in normal pitting, the site of the pit will give an acid reaction (pH is low as 3.4). This does not mean that acid caused the pitting—instead pitting caused the acid.

In other cases, the surface, (with the corrosion product in place) may be extracted with a small volume of hot distilled water, either by immersion or by repeated pouring over the specimen. The pH of the extract can then be determined.

**Detection of Color.** The corrosion products normally found on aluminum alloys are white to light or dark gray in color. The presence of colors or tints in the product indicate an abnormality that may give an important clue to the cause of corrosion. In one case a pinkish hue was detected in products on 3003 alloy conduit.<sup>6</sup> This indicated an oxidized state of manganese and suggested that the surface had functioned as an anode in a galvanic cell. This, coupled with the detection of acidity, led to the solution of the case described earlier.

A faint color of blue or green may indicate the presence of copper in the corro-

sion product and provide a clue to the cause of corrosion. An electrographic print of the corroded surface can be taken to confirm the presence of copper, and will provide the pattern of distribution on the surface. This may be found to be related to the corrosion pattern. In one case, rings of copper concentration were found around pits in the bottom of an aluminum kettle. These had been deposited by the local action currents from the pit, proving that the water being heated had been high in copper content, and provided the probable cause of pitting.

The corrosion products on copper-containing alloys such as 2017, 2024 and 2014 are frequently greenish in color and often provide the first clue to alloy identification. In this case of course no conclusions concerning copper in the environment can be drawn.

#### *Dissolution of the Corrosion Product From the Surface*

If a sufficiently large sample is available and if copper is suspected (for example, in the case of a pitted teakettle or domestic hot water tank) the amount of copper per unit area is determined from a 1:1 nitric acid leach of a measured area. As some data on surface copper concentrations have now been accumulated, an idea can be obtained of the severity of the condition.

#### *Removal of the Corrosion Product From the Surface*

If there is sufficient material for analysis, the corrosion product is brushed or scraped from the surface. If there appears to be more than one kind of product (based on color texture or structure) these should be separated carefully and analyzed individually. Examination of the product under a low power microscope may be helpful in such a separation.

#### Techniques Used to Determine the Composition on an Aluminum Corrosion Product

##### *Physical*

**X-ray Diffraction.** As indicated earlier, most aluminum corrosion products are amorphous to X-ray. However, in a few products definite patterns have been obtained. There is also the possibility that foreign crystalline matter may be present and that when identified this may give a clue as to the corrosive environment.<sup>5</sup> In addition, calcining the product for one hour at 1000°C may give good patterns. About 10 mg of sample is required.

**X-ray Fluorescence.** The X-ray fluorescence technique has had rather limited use to date in this laboratory because the equipment has only recently become available. The method is non-destructive and the equipment is highly sensitive to many elements. The maximum sensitivity on the equipment available is obtained with iron, whose atomic number is 26. The presence of elements below atomic number 12 (magnesium) cannot be determined.

**Differential Thermal Analysis.** Differential thermal analysis of aluminum cor-

rosion products has confirmed that they generally are of an amorphous nature. Irreversible exothermic reactions occur at temperatures of 400–600°C, which are indicative of phase transformations. This technique, therefore, has rather limited application in the study of corrosion products of aluminum.

**Electrographic.** Deposited metals more noble than aluminum can be dissolved anodically in contact with a gelatinized blotting paper to present a mirror image of the distribution of the original deposit. Specific chemical reagents are added to cause a coloration of the ions present.

**Spectrographic.** A 10-mg sample is required for spectrographic analysis using Harvey's semi-quantitative analysis procedure.<sup>7</sup> Because of the rapid rise in temperature when the DC arc is struck, it is advisable to calcine the corrosion product prior to analysis by this technique. Otherwise the water of hydration in the specimen is released violently and some or all of the sample may be expelled from the electrode. As already indicated, this method is semi-quantitative and is claimed to give results for some 43 metallic elements within a factor of 3 from the true chemical value. Experience in this laboratory has not confirmed these claims, especially in the early part of the program when calcination of the product prior to arcing was not carried out. Later data, obtained from calcined products has increased confidence in the technique to some extent. Perhaps more important is the high sensitivity of the method to most metallic elements; mercury in an alumina matrix is an outstanding exception. For this reason, a chemical concentration technique followed by spectrographic arcing has been devised for the determination of mercury in low amounts.

A recent refinement<sup>8</sup> for the spectrographic analysis of powders offers greater accuracy than is obtainable by Harvey's method if the Quantometer and standard samples are used. This technique has not been applied as yet to corrosion products, but will be applied in the future as there is no reason why it should not be successful. Spectrographic techniques have the advantage that only small samples are required to obtain a comprehensive analysis of the metallic elements.

**Microscopic.** When only small amounts of corrosion products are present on a sample, use may be made of a low power microscope (7X) to examine its nature. Such examination will reveal whether or not there is more than one kind present. It may suggest presence or absence of crystallinity, presence of uncorroded metal and give an idea of the color. Microscopic examination will reveal the difference between surface stains or smears and corrosion products. The nature of the corroded surface may give the experienced viewer an idea as to the nature of corrosion (i.e., chemical attack, intergranular corrosion, etc.). The microscope is also useful for carrying out micro spot tests with chemical reagents on small amounts of product as for example to

confirm the presence of aluminum, chloride, etc.

#### Chemical

**Qualitative.** Qualitative tests are usually made on the water-soluble fraction of the corrosion product. Conventional tests have been adapted for determination of chlorides, sulfates, carbonates, copper, iron and calcium. Testing also for phosphate and nitrate has been recommended.<sup>5</sup>

**Quantitative.** Quantitative tests have been developed for the determination of moisture, loss on ignition (LOI), pH, chlorides, sulfates, carbonates, calcium, copper, iron, magnesium, silicon, total aluminum and free aluminum in corrosion products. These methods are based on conventional procedures used for the analysis of aluminous materials.

#### Analytical Results

##### General

Sufficient material has been available in a few instances to enable a fairly comprehensive study of the corrosion product to be carried out. As this represents the first published attempt to fully characterize an aluminum corrosion product, all data obtained are reported, as is a brief account of the case history.

As the water content of aluminum corrosion products may vary from a very large value in the case of a gel freshly removed from an aqueous environment to a much smaller but still appreciable value in the case of an aged and free-flowing powder, a comparison of composition based on the sample weight will show wide variations on similar products which vary only as to the amount of free and combined water. To put the compositions on a comparative basis free of this variable the proposal is made to report the results as a proportion of the  $\text{Al}_2\text{O}_3$  content. Of course in the few cases where alumina is not the main component this cannot be done. It might be agreed that the constituents be reported as a proportion of the aluminum content of the product. However, as corrosion products sometimes contain unreacted

aluminum this is considered more open to confusion.

#### Corrosion Products Formed in Fresh Waters

**Swimming Pool Lined with Aluminum Sheet.** A private swimming pool was lined with 1100 alloy aluminum sheet. Water for the pool, pumped from a nearby spring, was fairly soft with total solids of 78 ppm and a total hardness of 35 ppm; the pH was 7.8. Copper was not detected in the water, (i.e., less than 0.01 ppm). Such water would not be expected to be corrosive to aluminum. Nevertheless, the sheet pitted within a few months after operation of the pool and formed nodules of buff-colored corrosion product. No explanation of the cause of corrosion could be determined in this case, but a summary of the data is presented to indicate the character of the product. X-ray diffraction techniques yielded no pattern either from the "as-received" sample or from the moisture-free sample (heated at 200°C for 1.5 hours). In other words the material was amorphous to X-rays. Upon calcination of the product (2 hours at 1000°C) a weak pattern indicated the presence of  $\text{Al}_2\text{O}_3$  and  $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$  (mullite).

Petrographic examination revealed that the product consisted of anhedral, angular particles 10-100 microns in size. At least two isotropic substances were present as well as traces of an anisotropic (crystalline) phase. The main isotropic constituent was colorless with a variable refractive index (R.I.) between 1.41 and 1.50 centering about 1.46. The second isotropic constituent was yellow with an R.I. of 1.51-1.53. Angular "Platey" fragments were colorless-to-yellow with an R.I. of about 1.50. It was concluded that the chief constituent was an alumina hydrogel, perhaps containing some silica, which has variable properties similar to those of certain minerals such as schrotterite. No material with the properties of gibbsite ( $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ ) was present.

The analytical results are given in Table 1.

The sum of LOI to 1000°C,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$  and  $\text{CaO}$  (assuming the  $\text{CO}_2$  to be present as  $\text{CaCO}_3$ ) is 100.9 percent,

which is a very good material balance. The above results were re-calculated as a percentage ratio of the  $\text{Al}_2\text{O}_3$  content and are given in this form in Table 2.

#### Aluminum Pipe in Cooling Trough

The water involved is soft (total hardness, as  $\text{CaCO}_3$ , 45-55 ppm) and is filtered, while the pH varies between 7.8 and 8.2. Some 99.5 percent aluminum piping corroded within six months after installation, but again no explanation could be offered as to why the metal corroded.

Petrographic examination showed that, as with the swimming pool product the particle size was 10-100 microns. At least two isotropic materials were present, the major one anhedral, and varying in color from transparent to yellow (R.I. 1.47). Thin plate-like fragments were present, as were some glasslike, transparent, isotropic fragments (R.I. 1.48). Traces of anisotropic material were also noted. As with the swimming pool product, the product was classed as a hydrogel. The chemical data obtained are given in Table 3.

The sum of these constituents totals 96.5 percent which is probably within the total experimental error. The constituent-to-alumina ratios are given in Table 4.

On comparing these ratios with those in Table 2, it will be noted that there is a greater amount of absorbed and combined water, perhaps because of its lesser age. The silica ratios are identical, whereas the  $\text{CaCO}_3$  ratio is higher in the swimming pool product.

#### Aluminum in Another Tapwater. A partial analysis of the corrosion product

TABLE 5—Composition of Corrosion Product of Aluminum Formed in London, England Tap Water

Constituent	Percent
Al	29.0
Si	0.76
Cu	0.1
Fe	0.15
Cl	Trace
SO <sub>4</sub>	Trace

TABLE 6—Composition of Corrosion Product Formed on 6061-T6 Sheet in Sea Water

Constituent	Percent
LOI at 105°C	6.7
LOI at 1000°C	55.0
Al (as $\text{Al}_2\text{O}_3$ )	41.6
Ca	0.7
Fe	0.03
Mg	0.45
Si	0.28
Cl	1.3
SO <sub>4</sub>	14.5
CO <sub>2</sub>	1.1

TABLE 7—Constituent/Alumina Ratios of 6061-T6 Corrosion Product Formed in Sea Water

Constituent	Ratio, Percent
Absorbed water	14.4
Combined water	80.0
SiO <sub>2</sub>	1.4
CaCO <sub>3</sub>	4.3

TABLE 1—Composition of Swimming Pool Corrosion Product

Constituent	Percent
LOI to 110°C	19.9
LOI to 140°C	22.5
LOI to 1000°C	50.9
Al (as $\text{Al}_2\text{O}_3$ )	43.4
Si (as SiO <sub>2</sub> )	4.3
Total C.	1.8
CO <sub>2</sub>	1.8
Na	0.03
K	0.05
Cl	Trace

TABLE 2—Constituent/Alumina Ratios for Swimming Pool Product

Constituent	Ratio, Percent
Absorbed water	46.5
Combined water	63.4
SiO <sub>2</sub>	10.0
CaCO <sub>3</sub>	9.3

TABLE 4—Constituent/Alumina Ratios for Cooling Trough Product

Constituent	Ratio, Percent
Absorbed water	55.4
Combined water	79.8
SiO <sub>2</sub>	10.0
CaCO <sub>3</sub>	6.0

**TABLE 8—Effect of Sea Water Velocity on Composition of Corrosion Products Formed in 99.5 Percent Al Tubing**

Pipe Diam. (in.)	3	2.5	2.0	1.5	1.0	0.75	0.5
	Percent*						
pH of extract.....	7.8	8.0	7.8	8.3	8.4	8.5	8.7
Absorbed H <sub>2</sub> O (1).....	7.4	4.2	5.2	7.2	9.3	9.4	8.3
(2).....	6.7	4.1	5.6	7.5	8.5	9.9	8.2
LOI at 980-1020 C.....	36.9	36.0	37.8	36.8	36.2	34.4	38.6
Total Al.....	5.5	3.9	3.9	5.9	18.8	18.0	11.1
Calc. Al <sub>2</sub> O <sub>3</sub> .....	10.4	7.3	7.3	11.3	35.5	34.0	20.9
Cu.....	0.03	0.03	0.04	0.10	0.12	0.17	0.23
Fe.....	1.6	1.5	1.4	2.1	3.5	4.8	6.3
Si.....	7.8	8.3	6.7	7.6	7.6	7.9	5.5
Ca.....	7.1	13.5	12.4	2.6	3.1	8.2	5.3
Cl.....	3.7	3.0	2.7	2.9	3.7	3.8	3.0
CO <sub>2</sub> .....	9.8	15.1	12.8	10.5	5.0	4.2	8.0
SO <sub>4</sub> .....	4.4	3.1	5.5	3.2	3.8	2.7	2.6

(1)—Dried at 110 C.

(2)—Karl Fischer Titration.

\* Except pH.

**TABLE 9—Constituent/Alumina Ratios for Sea Water Pipe Products**

Pipe Diam. (in.)	3.0	2.5	2.0	1.5	1.0	0.75	0.5
Constituent	Ratio Percent						
Absorbed water.....	71.	58.	71.	64.	26.	28.	40.
Combined water.....	154.	188.	235.	144.	52.5	50.0	92.3
SiO <sub>2</sub> .....	161.	243.	197.	144.	45.5	50.0	56.0
CaCO <sub>3</sub> (from Ca).....	170.	462.	425.	57.5	21.7	60.2	63.0
Cl.....	35.6	41.1	37.0	25.7	10.4	11.2	14.4
SO <sub>4</sub> .....	42.3	42.4	75.4	21.3	10.7	8.0	12.4

**TABLE 10—Miscellaneous Sea Water Corrosion Products**

Constituent	Material	
	6061-T6 Plate Percent	5052 Pipe Percent
pH of water extract.....	6.5	4.3
Absorbed water (110 C).....	12.0	21.3
LOI at 1000 C.....	49.6	53.5
Al.....	10.3	18.6
Al <sub>2</sub> O <sub>3</sub> (calc'd.).....	19.5	35.8
Si.....	1.2	0.18
Ca.....	1.1	0.18
Fe.....	0.27	7.7
Cl.....	0.9	6.9
CO <sub>2</sub> .....	3.5	0.28
SO <sub>4</sub> .....	11.3	16.0

was supplied by another laboratory. The material was amorphous to X-rays. The chemical data for the sample after drying at 110 C are given in Table 5.

In this case a material balance cannot be computed, since there are insufficient data. However, the silica-to-alumina ratio is only 2.9 percent or only one-third that obtained in the two preceding cases.

#### Corrosion Products Formed in Seawater

**Ship Grounding Plate.** A large 6061-T6 alloy sheet was used as an electrical ground on a wooden hulled vessel. This plate, which was not connected to the cathodic protection system, was covered with nodules of buff-colored corrosion product.

The chemical composition is given in Table 6.

The material balance of 98.9 percent is within the probable experimental error. It will be noted that the sulfate content is quite high. This may be due to concentration of sulfate on the alumina gel, that is corrosion product, via the mechanism of ion exchange. The magnesium present is probably due to the 1.0 percent Mg in the alloy, but may also have been exchanged, like the calcium, from the sea water.

The constituent/alumina ratios are given in Table 7. A comparison of these ratios with those previously noted shows that the combined water is identical to that of the cooling trough product mentioned earlier. The silica and calcium carbonate ratios differ quite markedly from the previous cases however.

**Seawater Velocity Test.** In a test at Harbor Island, N. C., seawater was pumped

through a series of ten foot long aluminum pipes (99.5 Al) for a period of three years. Prior to entering the aluminum system the water passed through other metal pumps and a short length of copper-nickel alloy tubing. The pipes varied in diameter from 3 inches to 0.5 inch and were arranged in order of decreasing diameter with the water entering the largest pipe and leaving the smallest. Due to other demands on the system, the velocity was not constant over the test period although of course the relative flow rates from diameter to diameter remained the same. On completion of the pipes were returned to the laboratory for determination of the extent of corrosion.<sup>9</sup> Considerable corrosion product was available in each length, and out of curiosity these were analyzed separately. The results are presented in Table 8.

There are several trends in the results which are presented without comment as no previous data exist for comparison and as there is no ready explanation for them.

(1) The pH of the water extract increased with decreasing pipe diameter (from 7.8 to 8.7).

(2) The copper content increased with decreasing pipe diameter (from 0.03 to 0.23 percent).

(3) The iron content was constant for the larger pipes (1.6 to 1.4 percent for 3, 2.5 and 2 inch pipe) and then increased at lower pipe diameters (2.1 to 6.3 percent).

(4) The silicon content was fairly constant regardless of pipe diameter (5.5 to 8.3 percent) and showed no trend.

(5) The calcium content increased as the pipe diameter decreased (7.1 to 13.5 percent in 3 and 2.5 inch pipes and then decreased at lower diameters varying somewhat from 2.6 to 8.2 percent).

(6) The chloride content was fairly uniform (2.7 to 3.8 percent) for all diameters.

(7) The carbonate content, like the calcium content, went through a maximum (15.1 percent CO<sub>2</sub> in the 2.5 inch pipe) and then decreased (from 12.8 to 4.2 percent) with the 8.0 percent CO<sub>2</sub> in the 0.5 inch pipe being an exception.

**TABLE 11—Constituent/Alumina Ratios for Miscellaneous Sea Water Product**

Constituent	Material	
	6061-T6 Plate	5052 Pipe
Absorbed water.....	61.6	59.5
Combined water.....	171.	70.0
SiO <sub>2</sub> .....	13.2	...
CaCO <sub>3</sub> (from Ca).....	14.1	1.3
Cl.....	4.6	19.3
SO <sub>4</sub> .....	58.0	44.7

**TABLE 12—Composition of Haiti Light-Tower Corrosion Product**

Constituent	Percent
LOI at 105 C.....	3.9
LOI at 1000 C.....	21.2
Al.....	46.0*
Cu.....	2.1
Fe.....	0.25
Mg.....	0.35
Mn.....	0.31
Si.....	0.31

\* Partly metallic.

TABLE 13—Alloy Compositions

	Element, Percent				
	Cu	Fe	Mg	Mn	Si
Composition on basis of corrosion product.....	4.6	0.54	0.76	0.67	0.67
Composition of cross-brace.....	3.89	0.33	0.52	0.60	0.37
Composition of 2017 alloy (nominal).....	4.35	0.3	0.65	0.60	0.45

(8) The variations in sulfate content (2.6 to 5.5 percent) showed no trend relative to pipe diameter.

The constituent/alumina ratios for absorbed water, combined water, silica ( $\text{SiO}_2$ ) and calcium carbonate (with the Ca values calculated to  $\text{CaCO}_3$ ) are given in Table 9.

**Miscellaneous.** Two other sea water corrosion products have been partially analyzed. One was taken from a 6061-T6 alloy plate immersed in Halifax harbor, while the other was taken from a pitted 5052 alloy pipe in a steel ballast tank on S.S. Sunrip. The results are given in Table 10.

There are some large differences in the composition which become interesting when compared to the environment in which they were formed. The 6061-T6 plate was corroding freely in sea water, while the 5052 pipe was in a steel tank probably grounded to the steel and thus functioning as an anode. The following may be pointed out:

(1) The pH of the water extract from the 5052 pipe product was quite acid (4.3) compared to the nearly neutral value (6.5) for the 6061-T6 plate, and compared to the alkaline values in Table 8 (7.8 to 8.7). As the environment around an anode tends to become acidic this is not unexpected and is evidence that the pipe was indeed grounded to the steel tank.

(2) The iron content of the pipe was high (7.7 percent) and was probably due to rust particles from the corroding tank wall which became embedded in the alumina corrosion product during its formation.

(3) The chloride contents differed markedly (0.9 vs 6.9 percent). The high chloride in the product on the 5052 pipe is further evidence that the pipe was acting as an anode since chloride ion tends to migrate toward an anode and the 6.9 percent figure is higher than was found in Tables 6 and 8.

The constituent/alumina ratios for these products are given in Table 11.

#### Corrosion Products Formed In the Atmosphere

**Haiti Light-Tower Structural Member.** After 21 years' service, a navigation light-tower was examined for corrosion of its aluminum components. They were found to be in excellent condition generally even though, from the point of view of present-day knowledge, an inferior alloy had been used. There was one location in which severe corrosion had occurred, however, that where the cross-brace members entered a concrete platform. At first only the corrosion product was available for testing; later, when the cross-braces

had been replaced, the corroded metallic sections were made available.

The composition of the corrosion product is given in Table 12.

Since this product was formed essentially by atmospheric corrosion (as compared with corrosion by immersion in liquid media) the elements reported above probably originated with the alloying constituents. In this case the constituent/alumina ratios were not calculated since the metallic aluminum content determined was not resolved into free and combined aluminum as would now be done. Instead the element-to-aluminum ratios were calculated and expressed as percentages to give the approximate alloy composition (Table 13). When the cross-brace members were made available for testing at a later date, the composition was determined. (The nominal composition of the closest standard alloy is also given for purposes of comparison.)

Thus corrosion products, formed in a manner such that exchange of metallic constituents is unlikely, can be used (with caution) to determine the alloy composition, when a sample of the metal itself is not available.

**Formed in Exhaust Air in Chemical Plant.** Two completely water-soluble samples of corrosion product formed on 5052 alloy sheet ductwork in an Ontario chemical plant had the analysis shown in Table 14. Considerable  $\text{SO}_2$  is present in the air in the vicinity of the ductwork. The high acidity values are also noteworthy.

**Miscellaneous.** Corrosion product data for a clad building sheet (7072 cladding) from (a) a British Columbia exposure and (b) an Ontario building near a railway, as well as data for the Alcoa 43 alloy statue on the Board of Trade Building, Chicago, Illinois, are given in Table 15.

The B. C. Kingstrong sheet (a) was corroded by mercury, as will be shown in a subsequent section. The high sulfate content in the Ontario building product (b) was no doubt due to the proximity of the building to the mainline railroad tracks where considerable  $\text{SO}_2$  is released by passing steam engines. The product from the Chicago Board of Trade Building Statue is interesting in that there is no correlation between its composition and that of the 43 type alloy from which it originated. It will be remembered that the Haiti Light Tower corrosion product composition presented in Table 13 had good correlation with that of the alloy from which it originated.

A corrosion deposit on the screening of a Toronto hospital presumably made from 5056 alloy was found qualitatively to contain considerable amounts of sul-

TABLE 14—Composition of Corrosion Product on 5052 Sheet Exposed in Ontario Chemical Plant

Constituent	Percent*	
pH of solution.....	3.0	2.2
LOI at 105 C.....	51.0	8.3
LOI at 900-980 C.....	4.0	39.0
Total Al.....	Absent	3.0
Trace Cl.....	Absent	....
$\text{SO}_2$ .....	52.0	60.0

\* Except pH.

fate, and only traces of nitrates and chlorides.

#### Presence of Copper in Aluminum Corrosion Product

When copper ions are present in a solution in contact with aluminum they tend to plate out on the surface in the form of metallic copper. Such areas of deposited copper are strongly cathodic to the adjacent aluminum surface and tend to cause pitting. When sufficient data have been accumulated it is hoped that the amount of copper found when compared to the known cases will be a valuable guide in deciding whether or not copper has been a factor in the corrosion found.

Table 16 gives the copper contents of several aluminum corrosion products in cases where copper was suspected to have been a cause of corrosion. Other miscellaneous copper values available have been included in the second part of this table for comparison.

In other cases, instead of determining the copper content of aluminum corrosion product separated from the metal, the surface has been leached with nitric acid and the amount of copper present expressed in milligrams per square foot. Again as more data of this type become available the significance of the amount found in any new case will be more apparent. The values obtained to date are given in Table 17.

#### Presence of Mercury in Aluminum Corrosion Products

Mercury ions tend to plate out on an aluminum surface and induce corrosion in a manner similar to that of copper. However, the effect is very much stronger with mercury than is the case with copper. Indeed if mercury can be detected in the corrosion product, it has probably been a major factor in the cause of corrosion. (The most sensitive spot test available for the detection of mercury is based upon its corrosive action on aluminum foil.<sup>10</sup> The test conditions are admittedly most adverse; the mercury is plated on the foil electrolytically and the limit of identification is claimed to be one part in ten billion. Under more favorable conditions it is known (as evidenced by the use of aluminum in the chemical process industry) that as much as a few parts per million mercury will not cause corrosion.) As mentioned earlier, corrosion due to mercury can sometimes be recognized by the appearance of the corroded surface.

Experience has shown that mercury is a rather infrequent cause of corrosion of

TABLE 15—Composition of Corrosion Products of Different Alloys Exposed in Various Industrial Atmospheres

Constituent	Material		
	7072 : B.C.	7072 : Ont.	43 : Chicago
pH of water extract.....	6.6	...	...
LOI at 110 C.....	...	...	4.8
LOI at 1000 C.....	...	19.7	26.3
Total Al.....	...	...	44.6
Free Al.....	...	...	27.0
Calc'd. Al <sub>2</sub> O <sub>3</sub> .....	...	37.2	33.3
Cu.....	0.02	...	1.3
Fe.....	0.008	...	0.72
Mg.....	...	...	0.36
Mn.....	0.2	...	...
Si.....	0.07	...	0.13
Zn.....	0.9	...	...
Pb.....	0.008	...	...
Cd.....	0.003	...	...
Cl.....	0.04	...	0.2
SO <sub>4</sub> .....	Absent	13.0	11.8
Color.....	Grey	...	...

TABLE 16—Copper Content in Aluminum Corrosion Products

Alloy	Case	Copper Percent
<b>(a) Copper Suspected</b>		
5056	Toronto hospital screening (copper flashing on building).....	2.82
99.5% Al	Unifin Tubing (moist copper dust in air).....	1.28
Unknown	Gas stripper in Montreal refinery (copper sulfate in system).....	0.36
Unknown	Piping carrying formaldehyde.....	0.20
<b>(b) Copper Unsuspected</b>		
2017	Haiti: Light-tower member (atmospheric).....	2.1
43	Chicago Board of Trade Status (atmospheric).....	1.3
7072	Hot water tank, tinned Cu element.....	0.42
7072	Hot water tank, stainless element.....	0.40
7072	Hot water tank, aluminum element.....	0.37
7072	Hot water tank, copper element.....	0.37
7072	Hot water tank from service.....	0.23
99.5 Al	Tests on rate of flow of seawater, 0.50 in. pipe.....	0.23
99.5 Al	Tests on rate of flow of seawater, 0.75 in. pipe.....	0.17
99.5 Al	Tests on rate of flow of seawater, 1.0 in. pipe.....	0.12
93.5 Al	Tests on rate of flow of seawater, 1.5 in. pipe.....	0.10
99.5 Al	Tests on rate of flow of seawater, 2.0 in. pipe.....	0.04
99.5 Al	Tests on rate of flow of seawater, 2.5 in. pipe.....	0.03
99.5 Al	Tests on rate of flow of seawater, 3.0 in. pipe.....	0.03
6061-T6	Electrical ground on wooden hulled vessel.....	0.03
EC	ACSR cable from Maritimes (atmospheric).....	0.03
6061-T6	Water storage tank anode.....	0.03
7072	Building sheet from Ontario plant near R.R. ....	0.02
3003	Embedded in concrete, Montreal Bldg. ....	0.01
3003	Embedded in concrete, Smith Falls, Ont., Hospital.....	0.004

TABLE 17—Copper Concentrations Found on Aluminum Surfaces

Article	Alloy	Description	Copper (mg/ft <sup>2</sup> )
Hot Water Tank.....	7072	Calgary, three years.....	570
Hot Water Tank.....	7072	Calgary, three years.....	450
Teakettle.....	3003	Kingston, Ontario, area.....	392
Hot Water Tank.....	7072	Sudbury, Ont., three years.....	370
Teakettle.....	3003	Shelburne, N.S., one month.....	249
Hot Water Tank.....	7072	Windsor, Ont., six years.....	210
Hot Water Tank.....	7072	Lethbridge, Alberta, 7 months.....	200
Piping.....	3003	Sugar refinery, 11 months.....	180
Teakettle.....	3003	Shawinigan, Que.....	171
Hot Water Tank.....	7072	Lethbridge, Alberta, 7 months.....	160
Hot Water Tank.....	7072	Calgary, Alberta.....	150
Hot Water Tank.....	3003	Lachine, Quebec, two years.....	130
Hot Water Tank.....	7072	Isle Malpeque, Que., 22 years.....	115
Hot Water Tank.....	7072	Toronto, 15 mos. (Cu element).....	100
Hot Water Tank.....	7072	Toronto, 3 years.....	65
Hot Water Tank.....	7072	Toronto, 10 months.....	65
Hot Water Tank.....	7072	Toronto, Ont., 6 years.....	50
Hot Water Tank.....	7072	Toronto, Ont., 6 years.....	40
Hot Water Tank.....	3003	Arvida, Que., anodized.....	10
Hot Water Tank.....	3003	Arvida, Que., anodized, 16 yrs. serv.....	6
Hot Water Tank.....	7072	Leamington, Ont.....	0.5
Hot Water Tank.....	7072	Kingston, Ont.....	0.3
Hot Water Tank.....	7072	Guelph, Ont.....	0.1
Seed Tank.....	1100		

\* Similar to 5086, but no chromium.

aluminum, due to limited presence in natural products, chemicals and foods. However, it may originate from an unsuspected source and in the solution of difficult pitting problems, it is well to consider an analysis for mercury. In addition, if corrosion has been rapid, and

if the appearance of the corrosion pattern or the corrosion product suggests mercury (this is quite rare), analysis for mercury should be carried out. On more than one occasion this has provided an unexpected answer to the problem.

The analytical determination of mer-

cury in trace amounts requires a special spectrographic technique and even then its detection is not always certain. However, if mercury is detected then the identification is positive. The mercury content of those corrosion products in which positive identification has been made to date are listed in Table 18.

### Discussion

(1) The more corrosion product composition data that is accumulated the better will be the chance that abnormalities in a new product will be recognized and their cause identified.

(2) The most common corrosion product found on aluminum is aluminum oxide with a large amount of water, some absorbed and some combined. This is a hydrogel system with no fixed compositions except in the case of very old or calcined products in which some crystalline material may appear. The product may contain varying proportions of chloride, sulfate, silica or carbonate or other constituents depending on its origin. Because of the large variation in the water content it is suggested that products be compared on an arbitrary constituent/alumina basis. This will not be possible in the few special cases which may be encountered in the chemical industry where the product is not primarily alumina, but a chemical reaction product such as aluminum acetate, chloride, sulfate, etc.

(3) In the use of aluminum corrosion product compositions to help determine the cause of corrosion, most success to date has resulted from consideration of the following items, and in roughly the order shown:

pH of surface or water extract

Mercury content

Copper content

Chloride content

Sulfate content

This is only a generality of course, since the probable cause can often be suspected from a review of the operating conditions and the presence of all the metals involved. In this case the composition of the corrosion product is used to confirm the suspicion.

The type of contamination differs depending on whether the environment is an atmosphere, or is aqueous, or solid. In cases of atmospheric corrosion, surface pH, chloride and sulfate contents provide the most frequent clue to the cause of corrosion. In aqueous environments copper is the most frequent culprit, followed by mercury. The pH of a water extract may also provide a clue as mentioned in the case of the ship's ballast tank. In solid environments such as concrete, wood, etc., surface pH often helps in solving the problem as do the chloride and sulfate contents.

(4) The finding of several percent of calcium and carbonate in aluminum corrosion products formed in waters was unexpected, and probably indicates the presence of calcium carbonate. This is

probably precipitated at the cathodic areas due to the increased pH. It appears at least in some instances that the assumed calcium carbonate content increases with the hardness of the water.

(5) The presence of silica in corrosion products formed in water was even more unexpected. It is possible that colloidal silica in the water may adsorb on the alumina product.

(6) The sulfate content of corrosion products formed in the atmosphere is believed to give an important clue to the corrosiveness of the atmosphere. It is known from experience that the atmospheric corrosion of aluminum is more rapid as the sulfur dioxide content increases and it seems probable that the sulfate in the product is in the form of aluminum sulfate. In the extreme case presented earlier (Table 14) the main corrosion product appeared to be aluminum sulfate. This compound has also been detected under artificial laboratory conditions with aluminum exposed to a humid atmosphere high in sulfur dioxide.

The mechanism is believed to be absorption of sulfur dioxide in the surface moisture film, its conversion to the trioxide and dissolution due to the sulfuric acid formed.

(7) The amount of chlorides in an atmospheric corrosion product may give a clue as to the cause of corrosion. In one case 0.5 percent chlorides was found in the product on aluminum stored near remelt furnaces where chlorine gas is used to flux the melt and is vented to the atmosphere. Other metal stored further away from the furnaces showed less corrosion and negligible chloride in the corrosion product.

(8) The value of determining the surface pH or pH of a water extract of the

Alloy	Mercury (ppm)
7072	6000
99.5%	2000
3003	400
7072	100
7072	20
7072	4
99.5%	2
7072	0.3
5032	0.27
5032	Present
....	Present
....	Present

corrosion product has already been emphasized and has provided an important clue in several case examinations.

(9) If the solution of a given corrosion problem is proving difficult it is well to analyze for mercury even though its presence is unsuspected or thought improbable. If it can be detected it probably was a major factor, while if it can not be detected there may still be some doubt as to whether traces were actually present.

(10) In cases of aqueous corrosion it is well always to determine the copper content of the corrosion product and/or the copper concentration on the surfaces since copper ions in solution are one of the most common causes of pitting of aluminum. It has been suggested<sup>11</sup> that for pitting to occur there must be present all four of the following:

- Oxygen
- Chloride
- Bicarbonate hardness
- Copper

Oxygen and chlorides are present in most waters and it is reasoned that bicarbonate hardness and copper are important variables. There is some evidence that more copper can be tolerated in a soft water than in a hard one. In analyz-

ing waters in corrosion cases it is the practice in this laboratory to determine:

- Total solids
- Total hardness
- Copper
- Chloride (sometimes)
- Sulfate (sometimes)

(11) The variation in composition of the corrosion product formed in seawater depending on flow conditions was unexpected. When more data of this kind become available the significance of the differences may become more apparent.

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## The Effect of Carbide Inclusions on Oxide Film Failure

# The Oxidation and Corrosion of Zirconium and Its Alloys\*

By B. COX\*

### Introduction

**I**N A STUDY of the effect of irradiation by fission fragments on the corrosion of Zircaloy-2, electron microscopy of the corroding surfaces was employed. The electron microscopy of corroding zirconium alloys was investigated also in the absence of irradiation, and several studies undertaken of the effect of specific metallurgical features on the course of the corrosion reaction. By this means it was possible to throw further light on the mechanism of film growth and breakdown and to provide comparative information for use in examining specimens corroded under irradiation. The results of the first of these investigations is reported here.

Carbon is reported to be one of the impurities in zirconium deleterious to corrosion resistance, the increased corrosion being thought to be due to preferential attack on the carbide phase<sup>1</sup>. The addition of tin to the alloy is said to counteract this effect of carbon. Carbon-melted Zircaloy-2, an alloy containing 1.5 wt. percent Sn, 0.1 wt. percent Fe, 0.1 wt. percent Cr and 0.05 wt. percent Ni, has a corrosion resistance only slightly inferior to that of arc-melted material (other impurities being present in about the same concentration in each). If the cause of the poor corrosion resistance in carbon melted zirconium is preferential attack on the carbide then it seems unlikely, since tin forms no carbide, that any appreciable dissolution of tin in zirconium carbide would take place. Hence it seems unlikely that the resistance of zirconium carbide to high temperature water and steam would be altered sufficiently to result in the considerable difference in corrosion resistance between the carbon melted zirconium and Zircaloy-2.

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TABLE 1—Analyses of Carbon-melted Zirconium and Zircaloy-2

	Zirconium Billet 1040	ZIRCALOY-2	
		Billet 793	Billet 887
Sn (Wt. %)		1.38	1.69
Fe (Wt. %)	0.035	0.10	0.10
Cr (Wt. %)	0.0025	0.07	0.10
Ni (Wt. %)	0.06	0.06	0.06
C (Wt. %)	0.06	0.16	0.12
O <sub>2</sub> (Wt. %)	0.085	...	...
N <sub>2</sub> ppm	35	165	245
H <sub>2</sub> ppm	...	15	...
Cu ppm	...	15	20
Al ppm	...	100	100
Mg ppm	...	<5	15
Mn ppm	...	7	10
Si ppm	...	155	<30
Ti ppm	...	...	<50

The carbide particles intercept only a small percentage of the metal surface; therefore breakdown of the oxide film must be a result of propagation outward from the nuclei provided by the carbide inclusions. The difference between the corrosion properties of carbon-melted zirconium and Zircaloy-2 then resolves itself into a difference in mode or speed of propagation from the nuclei present. The problem can therefore be reduced to the following: how and why does breakdown of the oxide film nucleate and propagate in carbon-melted zirconium, and what are the differences between this and the mode of breakdown in carbon-melted Zircaloy-2?

### Experimental

Material from three carbon-melted billets was used for this investigation; analyses are given in Table 1. Billet 1040 was of carbon-melted zirconium extruded in a copper sheath to 1.75 inch round bar and annealed; billets 793 and 887 were carbon melted Zircaloy-2 hot rolled sheet, not descaled, and 0.040 inch and 0.060 inch thick respectively. Specimens were of several different sizes depending upon the apparatus being used and surface preparation in any instance was by one of the following methods.

1. Metallographic polishing down to 3/0 emery followed by polishing on alumina laps.

### Abstract

The breakdown of the oxide films formed on carbon melted zirconium and Zircaloy-2 has been studied using optical and electron microscopes. The nature of the breakdown process is illustrated and an explanation is offered for the effect of tin, iron, chromium and nickel alloying additions on corrosion resistance of carbon melted zirconium.

3.2.3

2. Abrading to 3/0 emery followed by attack polishing<sup>2</sup>.

3. Cathodic bombardment etching with argon ions following surface preparation by attack polishing<sup>3</sup>.

4. Abrading to 3/0 emery followed by pickling in HF/HNO<sub>3</sub> solution.

Specimens were corroded in 300 C sodium sulfate solution using small 3/4-inch ID titanium autoclaves<sup>4</sup>, and in 300 C steam at atmospheric pressure. Corrosion was followed by weight change and visual observations were made at intervals using both light microscope and a Philips EM.100 electron microscope. Replicas were prepared by a two stage formvar-carbon method and were shadowed with gold-palladium alloy at an angle of about 30 degrees. The results of the weight change measurements are shown in Figure 1. The use of titanium high pressure equipment throughout ensured the maximum possible cleanliness of specimen surfaces.

Small specimens of carbon melted zirconium were examined directly in the electron microscope by the method de-

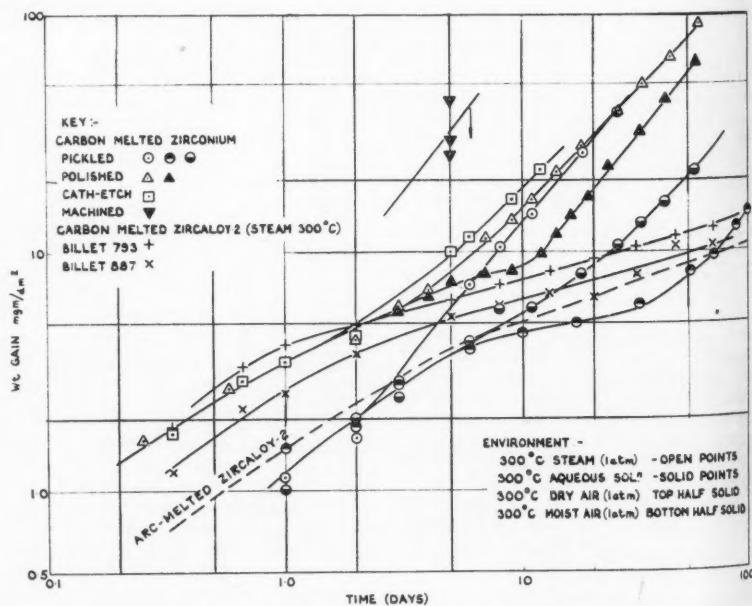


Figure 1—Corrosion of carbon-melted zirconium and Zircaloy-2.

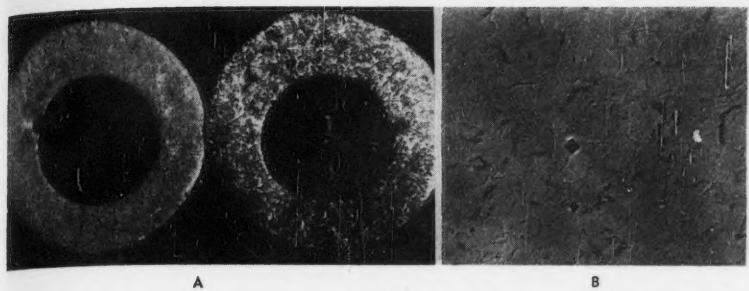


Figure 2—Enlarged view of test specimens. Photograph A at left shows typical specimens for direct observation under the electron microscope, approximately 15X; photograph B at right is micrograph of carbon-melted zirconium (cathodically etched). The microhardness indents assist in the location of particular inclusions during electron microscopy. Approximately 150X.

scribed by Edwards and Pickering<sup>6</sup>. These specimens were machined to 0.080 inch dia. x 0.020 inch thick with a central hole 0.020 inch dia. and were then chemically milled down to a thickness not greater than 0.003 inch using hot HF/aqua regia solution. The carbide particles were left in relief by the pickling. It was possible to examine these particles directly both before and after corrosion by viewing the edge of the central hole in profile; thus an electron shadowgraph of the surface was obtained. The edges of these specimens became rounded as a result of the pickling and were of sufficient thinness for all oxide growths on the inner edge to remain in focus at magnifications up to ~10,000 X. An enlarged view of two of these specimens is shown in Figure 2. The notch on the inner edge was used to locate specific points on the surface for subsequent examination.

### Results

The carbides in all three materials were similar in appearance and size (Figures 2 and 3) and effects due to the

distribution of the carbides were not expected to be significant. Tufts of white oxide became visible on the surfaces of all specimens after very short exposure times and as a result replication for electron microscopy was difficult. As some straining of replicas resulted when very rough surfaces were being examined a check for reproducibility was made by replication of the same area several times. On fresh surfaces no differences were apparent on different replicas; with the rough corroded surfaces some small differences were apparent (Figure 4) but these are not so great as to raise any problems of interpretation. As the kinetics and nature of the corrosion process were found to be dependent on surface preparation, the results for different materials prepared by the various methods will be considered separately.

### Carbon-melted Zirconium

The optical micrographs of this material (Figure 2b) showed that the

material consisted of uniform almost equiaxed grains with carbide particles lying at the grain boundaries. No other precipitated phases were detected either at grain boundaries or within the grains.

### Polished Specimens

As polished, these specimens were covered with a uniform thin Beilby layer so that individual grain structure did not appear under polarized light. The sites of carbide inclusions were usually visible but in most instances the sharp boundary of the carbide was not visible. After only one day's corrosion of these specimens, tufts of white oxide were visible at the sites formerly occupied by the carbide inclusions. The intervening film showed a uniform interference color apart from bands of higher order colors which apparently radiated from the carbide sites (Figure 5a). As a result of further corrosion these bands developed a thicker oxide film than the rest of the surface and became progressively grey and then white under dark field illumination (Figure 5b). The implication of this is that as the film thickens, white oxide is forming in ridges underneath the protective film. As these ridges became progressively whiter this oxide breaks through the protective film and no contrast between the ridges and the nuclei is then observed.

Electron microscopy showed the nuclei clearly (Figure 6a) but failed to show up the ridges apart from faint indications at low magnification due to shadowing contrast (Figure 6b). At longer corrosion times cracking was observed to spread outward from the nuclei along these ridges (Figure 7a); this stage cor-

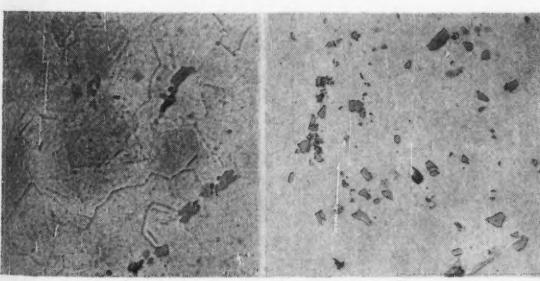


Figure 3—Carbide inclusions in zirconium and Zircaloy-2. Photograph A shows inclusions in zirconium which appear in Figure 8. Magnification is approximately 450X. Photograph B shows carbide in Zircaloy-2 (billet 793), magnification approximately 250X.

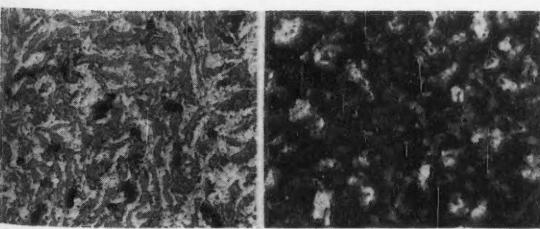


Figure 5—Micrographs of polished specimens of carbon-melted zirconium. Photograph A shows thicker oxide forming in "worms" after 1 day's corrosion, approximately 120X; photograph B shows the subsequent development of this pattern during breakaway (7 days), approximately 90X.

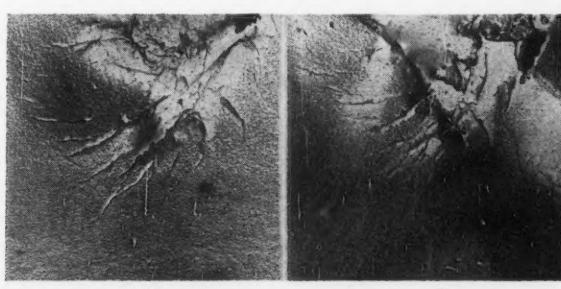


Figure 4—Electron micrographs of the same area on two different replicas. The variations from replica to replica due to straining during removal from the metal surface are of a relatively minor nature. The magnification was nominally the same but in practice quite considerable variations are found. Both micrographs have magnifications of approximately 2000X.

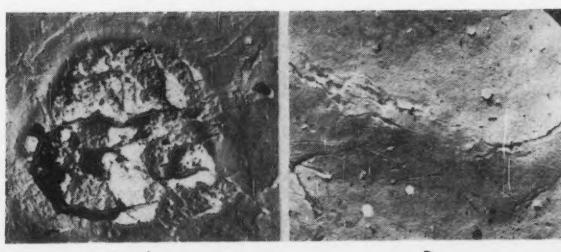


Figure 6—Electron micrographs of carbon-melted zirconium (polished). Photograph A shows white oxide nucleus formed at an early stage of corrosion (1 day), approximately 2200X; photograph B is a low magnification (1100X) picture showing a ridge (see Figure 5) with cracking along the top (six days). The ridge is visible only by virtue of the small change in contrast of the shadowing on either side of it.

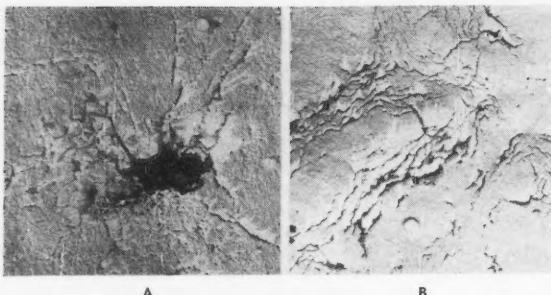


Figure 7—Electron micrographs of carbon-melted zirconium (polished). Micrograph A: cracking spreading outward from a nucleus (3 days), approximately 2000X. Micrograph B: a later stage in the cracking of the oxide (5 days), approximately 1750X.

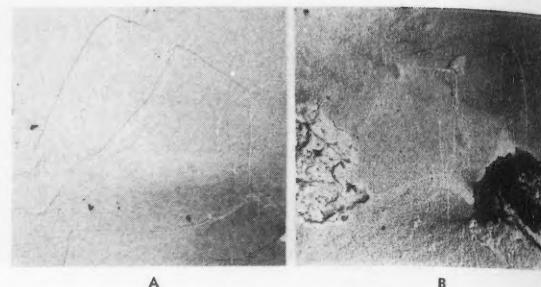


Figure 8—Electron micrographs of two carbide inclusions visible in Figure 3. Micrograph A: after cathodic vacuum etching, 1750X; photograph B: after eight hours corrosion at 300 C, 1750X. The two micrographs are mirror images.

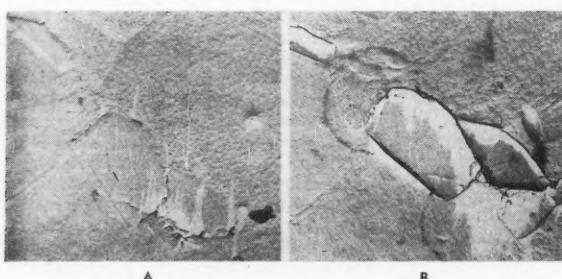


Figure 9—Initial attack on metal adjacent to the carbide precipitate. Micrograph A: group of carbide inclusions after 15 minutes in steam at 300 C and 1 atmosphere; micrograph B: the same group of carbides after one hour under the same conditions. Both micrographs approximately 1650X.

responded to the region where the ridges were becoming whiter under the light microscope. Subsequently cracking spread rapidly along the ridges and spread to the intervening oxide film (Figure 7b), which still showed a 3000-3500 Å interference color at this time. Henceforward satisfactory replication of the surfaces could not be obtained and a reflection microscope was not available; however, shadowgraphs of the surfaces of pickled specimens enabled subsequent events to be followed.

The course of these ridges of thick oxide is unrelated to the grain structure of the metal; this latter showed up at an early stage in oxidation as the Beilby layer was probably very thin (Figure 5a). The formation of these ridges and their positions probably are attributable directly to the method of specimen preparation employed.

#### Attack Polished and Cathodically Etched Specimens

Visual examination of these specimens showed that the oxide nuclei were formed very rapidly. Complete disappearance of the carbides took place in less than six hours on cathodically etched specimens. No formation of ridges was found in these specimens but a more uniform graying of the film spreading outward from the nuclei was found. These observations were confirmed by electron microscopy. The size of the nucleus formed after only eight hours was very much larger than the initial carbide inclusion (Figure 8). This suggested that the area around the inclusion had low corrosion resistance; cracking spread outward uniformly from these

inclusions without any preference for grain boundaries or other obvious directions although the oxide was usually too rough for replication at this stage.

The first hint that nucleation was due to attack on the area surrounding the inclusion and not on the inclusion itself came from the examination of carbon melted Zircaloy-2 specimens. Subsequent examination of zirconium specimens after very short exposures (15-60 minutes) confirmed this (Figure 9). It was also noted after short exposure times that interference color films, only slightly thicker than on the metal, formed on the carbide particles. After slightly longer periods of attack the carbide appeared to be surrounded by a white halo and the fact that the inclusion disappears after longer exposures may not be due to its complete oxidation. It may even be crushed as a result of the stresses resulting from the growth of the oxide around it and the high Pilling-Bedworth ratio (1.56). The small cracks observed in some carbide inclusions after cathodic etching are thought to be due to thermal stress on the carbide as a result of the heating of the sample during etching and the resulting thermal expansion.

#### Pickled Specimens

Pickling of carbon-melted zirconium results in pitting around the sites of carbide inclusions; the remainder of the surface is left in a smooth condition with the grain structure clearly visible. The material removed from around the inclusions during pickling is thought to be the low corrosion resistant material and hence the observation that pickled specimens show the lowest weight gains is not

unexpected. Breakdown of the oxide film on pickled specimens eventually takes place but the cracking in this case often follows grain boundaries as a result of preferential oxidation along them. Where the less resistant material has not been removed completely, formation of nuclei and more general cracking still take place (Figure 10). The pickle does not attack the carbides themselves but in most cases they fall out as a result of attack on the surrounding metal. An electron shadowgraph of a carbide inclusion on the verge of falling out is shown in Figure 12a. In cases where the carbide is still present owing to incomplete removal of the surrounding metal of low corrosion resistance it is possible to see the unattacked carbide quite clearly after much longer exposure times than in the case of polished or cathodically etched material (Figure 11a). This observation adds weight to the contention that the attack is not on the carbide phase but on the metal surrounding it. At longer periods when satisfactory replicas could not be obtained from specimens the course of the breakaway could be followed from electron shadowgraphs of pickled specimens. These showed that, after cracking of the oxide film, crystals of  $ZrO_2$  grew out through the cracked region (Figure 13). The tufts of white oxide formed at inclusion sites were also visible in profile and during cracking semi-transparent oxide films were observed which gave the impression that the thin protective film was curling back along the lines of the cracks (Figure 14).

*Carbon-melted Zircaloy-2*  
Examination of attack polished speci-

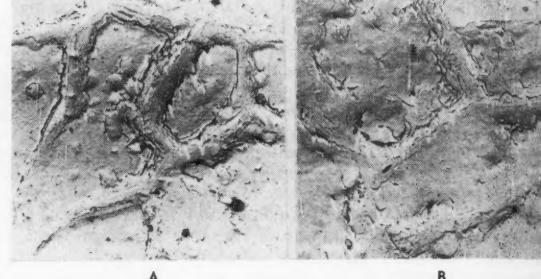


Figure 10—Electron micrographs showing the failure of the oxide film on pickled zirconium specimens. The ridges formed along grain boundaries and the cracking of the oxide are clearly visible. Micrograph A is at 1000X and B at 2000X.



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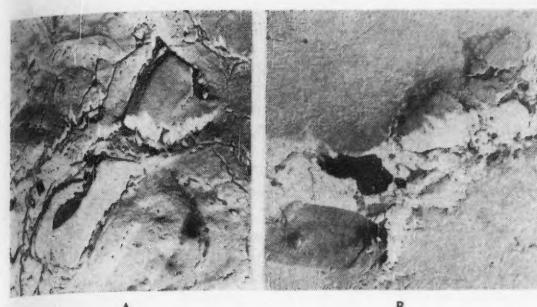


Figure 11—Electron micrographs showing carbide particles still visible in zirconium (pickled) after eight days (micrograph A, 2000X) and in Zircaloy-2 (attack polished) after eight days (micrograph B, 3000X).

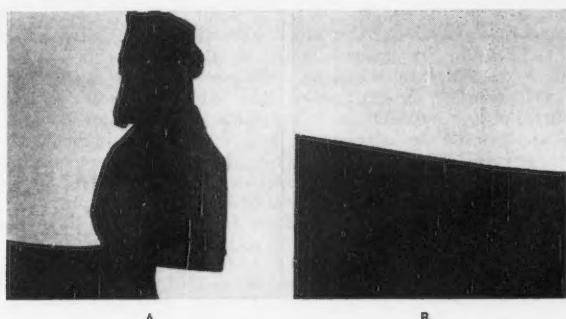


Figure 12—Electron shadowgraphs of carbon-melted zirconium. Micrograph A shows carbide particle before corrosion, 2000X; micrograph B shows surface of pickled specimen initially, 5000X.



Figure 13—Electron shadowgraph of the zirconium surface after breakaway (10 days), 950X.

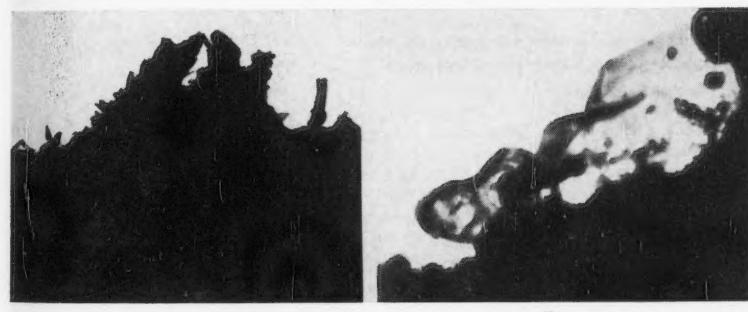


Figure 14—Electron shadowgraphs of zirconium surface after breakaway. Micrograph A: tuft of white oxide formed at the site of a carbide particle, 1250X; micrograph B: thin oxide film curling back after cracking, 650X.

mens from billets 793 and 887 soon indicated that the initial attack took place on the area immediately surrounding the carbide inclusion. These areas appeared to be surrounded by white haloes when viewed under dark field illumination. Attack on these areas was much slower than in the case of the carbon-melted zirconium specimens and carbide inclusions were still clearly visible after 20 days oxidation with only a small region of white oxide surrounding them (Figure 11). Electron microscopy confirmed these observations and showed that there was far less attack on the surrounding area than was found on carbon-melted zirconium. Billet 793, which has a low nitrogen content by British standards, and billet 887 with a high nitrogen content show corrosion rates almost identical with those found for arc-melted Zircaloy-2. Total weight gains are very little higher than those of arc-melted material at equivalent times and are less than the figures quoted by Thomas<sup>1</sup> for American arc-melted Zircaloy-2.

The observation that the area susceptible to corrosive attack by high temperature solutions or steam is the area immediately surrounding the carbide inclusions, and that the inclusions themselves are not seriously corroded, provides an explanation for the effect of different surface preparation on corrosion of carbon-melted material. A polished specimen in which this material lacking in corrosion resistance has been smeared out over the surface gives rapid breakdown of the oxide film. The low-resistance metal is smeared out in bands of material which oxidize more rapidly than the surrounding area; this leads to the growth of ridges of thicker oxide along these bands and cracking of the oxide film. This cracking spreads rapidly as a result of stress relieving and rapid oxidation at the base of cracks. It leads to complete failure of the protective film.

Samples which have been attack polished, cathodically etched or machined with a sharp tool do not have metal

from around the inclusions spread over the surface. Rapid severe attack on these areas with penetration into the metal via the accessible material of low resistance results in severe stressing of the oxide surrounding the tufts of white oxide which are formed. This leads to radial cracking outward from the nuclei present and to eventual failure of the whole oxide film.

Pickling of carbon-melted zirconium results in pitting around the carbide inclusions as a result of preferential dissolution of the poor material. This leaves mainly metal of good corrosion resistance exposed and the corrosion rates found for pickled specimens correspond closely with those for arc-melted zirconium, and for samples of carbon-melted zirconium oxidized in dry air (in which preferential attack on the areas round the carbides does not take place). Thus pickling of specimens apparently results in improved corrosion resistance. However, all the poorly resistant material is seldom removed by pickling and where this material is still present the course of events outlined above takes place.

On parts of the surface where fairly complete removal of poorly resistant metal has taken place, breakdown of the oxide film occurs by preferential oxidation and cracking along the grain boundaries. This grain boundary cracking may be due to enhanced diffusion through the oxide in these areas as has been suggested in the case of arc-melted zirconium,<sup>6</sup> or it may be due to the concentration of impurities, in this case carbon, along these boundaries. In view of the fact that a large concentration gradient of impurity atoms is obviously present in the area surrounding the carbides and that these tend to precipitate in grain boundaries anyway, the latter explanation would seem to be the more probable explanation in this instance. This grain boundary failure is observed only where the metal with low corrosion resistance has been pickled out. In other instances general cracking of the film occurs before this effect becomes prominent.

In the case of carbon-melted Zircaloy-2 the volume of material with low corrosion resistance surrounding inclusions is very much less than that found with carbon-melted zirconium. The oxide films appear to be able to withstand the stresses

imposed by the oxidation of this small amount of surrounding metal without cracking and the ultimate failure of the oxide in this case is due to a different mechanism. It is suggested that the metal of low corrosion resistance which is found around the inclusions in both arc-melted zirconium and Zircaloy-2 is otherwise similar to the bulk of the material apart from a high concentration of carbon in solution. The action of tin (and perhaps also iron, chromium and nickel) as an alloying element may be to reduce the solubility of carbon in zirconium and hence to reduce the area around each inclusion which is susceptible to attack. Tin does not form a carbide itself and it seems unlikely, therefore, that the tin would dissolve to any extent in the actual inclusion.

Thus the presence of the alloying elements in carbon-melted Zircaloy-2 results in material which has a corrosion resistance at 300°C almost equivalent to that of arc-melted Zircaloy-2. The slightly higher initial weight gain is due to the oxidation of the small amount of material present with low corrosion resistance. In the case of zirconium, increasing carbon content has been found to result in progressively poorer corrosion resistance.<sup>1</sup>

The reason for this appears not to be attack on the carbide inclusions present but on the region surrounding them containing a high concentration of carbon in solution. Failure of specimens in which this material has been largely removed by pickling is still rapid and in this case high concentration of carbon in solution along grain boundaries seems the most likely explanation of the type of failure found in these cases.

### Conclusion

Breakdown of the oxide film on carbon-melted zirconium originates at areas of low corrosion resistance surrounding the carbide precipitate and not at the carbides themselves. The subsequent failure of the film can be explained on the basis of stresses set up as a result of this rapid attack. From this the behavior of material prepared in different ways can be anticipated by considering the condition in which the areas around the inclusions would be left by the preparation method. Thus, if these areas are pickled out the rapid nucleation and failure does not take place and preferential oxidation along grain boundaries perhaps due to concentration of carbon along them leads to cracking and failure of the film. The

effect of the alloying elements in carbon-melted Zircaloy-2 results from their influence in reducing the areas surrounding carbide particles which are susceptible to rapid corrosion, and which may correspond to regions of high carbon content in solution. The stresses in the oxide in this instance are not sufficient to cause the propagation of cracking over the surface.

### Acknowledgment

The author would like to thank Mrs. B. C. Jenkins for the preparation of replicas, R. Harwood for the preparation and D. Armstrong for the cathodic vacuum etching of specimens. The advice of J. Cooley on electron microscopy and the interest shown by Dr. J. K. Dawson in this work are appreciated.

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**Any discussion of this article not published above will appear in the December, 1960 issue.**

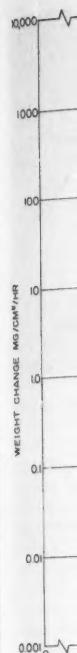


Figure 1—R...



Figure 2—

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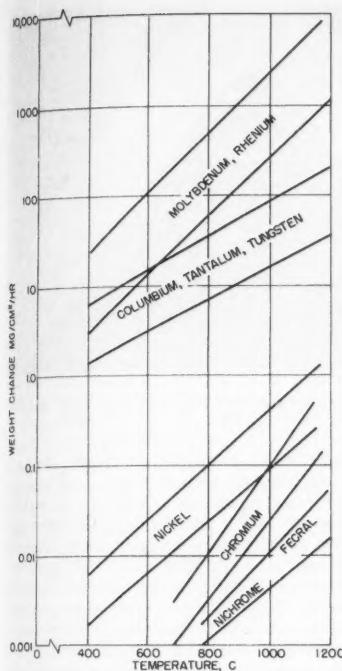


Figure 1—Relative oxidation behavior of metals and alloys for high temperature service.

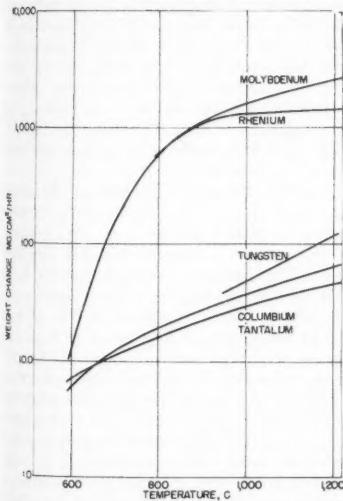


Figure 2—Approximated air oxidation relationships for refractory metals.

## Oxidation Behavior and Protection Of Refractory Metals\*

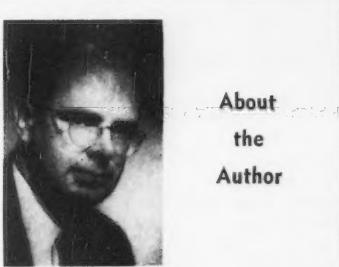
By CHESTER T. SIMS

### Introduction

THE STRENGTH of most unalloyed metals has a relationship with either their melting point or their transition temperatures. Since most of the highest melting metals are isomorphous from room temperature to their melting point, strength at high temperatures is good compared to metals melting at lower temperatures. This has created tremendous interest in the so-called refractory metals for use as construction materials. However, strength is not the only consideration needed for high temperature service. Oxidation resistance is also important in many applications, and most of the truly refractory metals have notably poor oxidation resistance. This article will review the overall oxidation behavior and discuss some recent developments in oxidation prevention of the refractory metals.

The easiest way to protect a metal from oxidation is by making use of its stable oxides as barriers. Many of the low-melting common metals such as aluminum, lead, magnesium, and indium possess oxides that melt at considerably higher temperatures than the parent metal. The relatively refractory oxide often assists oxidation resistance. Conversely, truly high-melting metals such as tungsten, rhenium, tantalum, molybdenum, osmium, ruthenium, and columbium form oxides which either melt or decompose far below the melting point of the parent metal (See Table 1). This relative physical instability of oxides of refractory metals has seriously hampered their wide-spread use in air at elevated temperatures. Tremendous effort is being exerted to study the properties of these metals and their oxides more closely,

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### Abstract

Relative oxidation behavior and degree to which oxide properties affect the oxidation resistance of columbium, tantalum, molybdenum, tungsten and rhenium are presented. Recent work in development of oxidation-resistant columbium and tantalum alloys is reviewed and the current status of protection of molybdenum by cladding and coating discussed.

and to develop methods to overcome the disadvantage brought about by lack of physical stability in the oxides.

### Oxidation Behavior of the Refractory Metals

In Figure 1, 2, 3, 4, 5, 6, 7 oxidation rates for the various refractory metals are outlined.\* The figure is meant to give only rough comparisons, not to show modes of oxidation. An extremely wide range of behavior exists. For instance, molybdenum oxidizes approximately five times as readily as either unalloyed nickel or chromium. The advantage to be gained by coating or cladding molybdenum with an oxidation-resistant material like Nichrome or Ferale for service at 1000 C is obvious from this chart.

Figure 1 also emphasizes the appreciable difference in behavior between those refractory metals which form vola-

\*The data for Mo and Re is derived from weight-loss figures, the other materials weight-gain; for the purposes of this article, the relatively small correction possible has not been applied.

TABLE 1—Properties of Refractory Metals and Their Oxides

Metal	Melting Point, Degrees C	Known Oxides	Melting Point, Degrees C	Boiling Point, Degrees C	Oxide-to-Metal Volume Ratio
Columbium.....	2380	CbO CbO <sub>2</sub> Cb <sub>2</sub> O <sub>5</sub>	(>1440) (>1440) 1440	..... Near M.P.	1.37 1.87 2.69
Molybdenum.....	2630	MoO <sub>2</sub> MoO <sub>3</sub>	(>795) 795	Sublimes	1.94 3.24
Tantalum.....	3000	TaO <sub>2</sub> Ta <sub>2</sub> O <sub>5</sub>	..... 1873	.....	2.73
Rhenium.....	3180	ReO <sub>2</sub> ReO <sub>3</sub> Re <sub>2</sub> O <sub>7</sub>	(>297) (>297) 297	..... 363	2.16 3.38 6.65
Tungsten.....	3440	WO <sub>2</sub> WO <sub>3</sub>	1580 1470	(1750)	2.08 3.35

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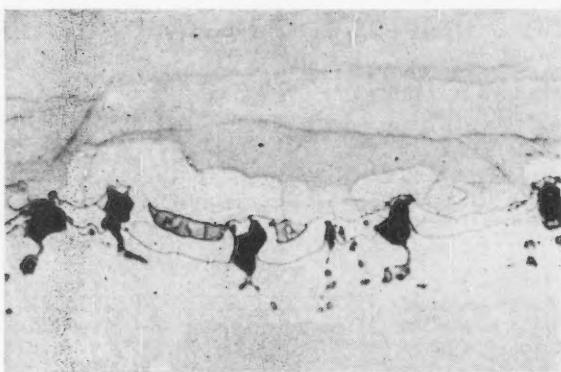


Figure 3—Nickel-molybdenum interface in nickel-clad molybdenum showing intermetallic layer and diffusion voids. Approx. 280X.

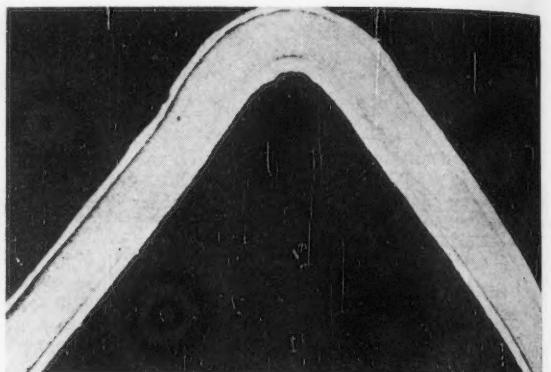


Figure 4—Section of clad molybdenum sheet showing bend ductility. Approx. 22X.

tile oxides at service temperatures from those refractory metals which form solid oxides. This behavior is delineated further in Figure 2, where approximate air-oxidation rates at 1000°C are plotted for the five best-known refractory metals. Molybdenum and rhenium, both possessing fluid oxides, react at a much greater rate than materials with solid oxides. The divergence of tungsten from behavior characteristic of a metal with a solid oxide toward that of a metal with a fluid oxide in the range 1000-1200°C is also apparent.

In addition to the fact that their oxides may melt below proposed service temperatures, other factors also promote poor oxidation resistance in refractory metals. One of the most obvious is the oxide-to-metal volume ratio which is usually unfavorable,\*\* so that the solid metal has difficulty in forming an adherent integral oxidation-resistant coating. It is apparent from Figures 1 and 2 that the refractory metals can be divided into two loose groupings.

#### Solid Oxide Formers

Columbium, tantalum, and tungsten (up to 1200°C) possess oxides that melt at moderately high temperatures (See Table 1), although other properties such as oxide-to-metal volume ratio may be poor. Moderate changes in the properties of these oxides, such as those that can be produced by alloying the base metal, can be expected to affect oxidation behavior.

#### Fluid Oxide Formers

Molybdenum, rhenium, osmium, ruthenium and tungsten (above 1200°C) form molten or gaseous oxides at temperatures of interest. Chances for improving the oxidation resistance of these metals by alloying the base metal is much less unless a completely new kind of oxide can be formed on the metal, such as nickel molybdate ( $\text{NiMoO}_3$ ) which can be created on molybdenum-nickel alloys. Thus, on the basis of these rather simple considerations, one group of refractory metals seems to show promise for devel-

opment as oxidation-resistant materials, while another group shows poor potential. Obviously, all of these metals potentially can be protected by oxidation-resistant barrier materials applied by cladding, coating or spraying, regardless of the characteristics of the base-material oxides.

#### Recent Progress Toward Oxidation Resistance

Currently, technology in the United States is directed toward both obvious aspects of this situation. Considerable effort is going into development of oxidation resistance in refractory metals by alloying of the base metal. Meanwhile, considerable advances are being made to provide oxidation resistance by application of suitable barrier coatings or claddings. An attempt will be made to review the state of the art individually for each refractory metal.

#### Molybdenum

The metallurgical technology of molybdenum metal is well ahead of the other refractory metals. Molybdenum is relatively light, strong, cheap and available in quantity on the North American continent. However, its oxidation resistance is low and attempts to cause improvement by alloying have been generally unsuccessful.<sup>8</sup> Stabilization of the normal oxide,  $\text{MoO}_3$ , by straight-forward alloying has been unsuccessful with a single important exception. Molybdenum-nickel alloys develop a double oxide,  $\text{NiMoO}_4$ , which is refractory and oxidation resistant at normal service temperatures.<sup>9</sup> Unfortunately, nickel molybdate spalls when thermally cycled. Another disadvantage is that molybdenum-nickel alloys have poor mechanical properties. Thus, it is evident that molybdenum must be protected from oxidation by either coating or cladding with an oxidation-resistant material, discussed in some detail below.

In cladding of a refractory metal such as molybdenum,<sup>10</sup> the major objective is to place an oxidation-resistant metal or alloy layer on the molybdenum, between it and the ambient oxidizing atmosphere. A number of factors must be considered when the cladding materials are selected. Since most of the cladding materials being considered today are based on metals that are low-melting

relative to molybdenum (Nichrome, Inconel) the phase relationships of systems involved must be carefully considered because of the possibility of excessive diffusion at service temperatures. Systems which form an excessive number of eutectics and compounds, such as shown in Figure 3, must be avoided. The presence of eutectics increases the diffusion rates, usually of the cladding components into the core material, causing a decrease in the effectiveness of the oxidation-resistant cladding. Diffusion voids and compounds result in poor clad-to-core bonds. An ideal clad-to-coating situation would be one in which the cladding alloy and the base material are isomorphous and do not diffuse together rapidly. Many problems can be avoided if a series of interlayers are selected so that isomorphism from the cladding metal to the core metal is created.

A clad refractory metal can be made by a number of hot fabrication processes, all of which are based on the concept that any two clean metals will bond if placed in intimate contact under a high vacuum and heated. For instance, a molybdenum core consisting of a rectangular plate can be placed in a picture frame of oxidation-resistant material. This assembly is placed between cover plates of the oxidation-resistant material. Following edge-welding and evacuation, it then can be hot rolled to a ductile oxidation-resistant refractory metal. A cross-section of a typical molybdenum sheet is shown in Figure 4. Clad rod, wire and some simple forged forms also can be manufactured.

When clad sheet is cut up into small sections for service, such as for nozzle guide vanes in jet engines, edge protection must be considered. Oxidation-resistant wires are laid into a trough formed at the edge (by undercutting the exposed molybdenum with hot caustic) and a weld bead laid around the edge to provide protection.

In selection of cladding alloys, reaction products of the cladding material not only must be compatible with the core material, but also with reaction products of the core metal and oxygen. For instance, molybdenum trioxide attacks iron and ferrous alloys very aggressively. Even a pinhole through a ferrous cladding on molybdenum will allow mo-

\*\*Generally, an oxide:metal volume ratio slightly greater than one is expected to be favorable for good oxidation resistance by forming a tight scale. However, refractory metals have oxide:metal ratios so high that the oxide buckles and cracks away from the surface as soon as it forms.

lybdenum trioxide to form and flux the coating oxide causing very rapid disintegration of the entire clad body. On the other hand, as discussed above, nickel forms a refractory double oxide with molybdenum. Nickel base claddings on molybdenum produce a self-healing nickel molybdate scale over a pinhole. Thus, nickel-base claddings not only act as barriers to oxidation attack, but generate sacrificial protection at imperfections in the coating. (See Figure 5)

Cladding is one of the most effective but high-cost methods of protection for refractory metals. Clad molybdenum sheet is being widely utilized in the jet engine field on an experimental basis. The present projected maximum service temperature is about 1200 C. This temperature is limited by the adhesive strength of the cladding materials, since most of those considered melt at temperatures between 1300 and 1600 C. Claddings of interest for the future may be built on oxidation resistant columbium or tantalum base alloys.

Coatings applied to molybdenum are receiving even more attention than claddings. They can be applied to both regular and complicated shapes, such as might be produced by forged or cast molybdenum. Coatings are usually applied by spraying, electroplating, replacement, or dipping.

Certain coatings are applied by flame-spraying. These are various combinations of aluminum, silicon, chromium, nickel and boron. Typical alloys are aluminum-silicon-chromium, nickel-chromium-boron, and nickel-silicon-boron. Flame-sprayed coatings have moderate ductility at high temperatures. Their principal disadvantage is excessive brittleness at low temperature or at room temperature, so they are considered promising for static service only.

Molybdenum disilicide ( $\text{MoSi}_2$ ) coatings are also of interest. These are applied by a paint-and-sinter method or by vapor deposition onto the molybdenum surface. These coatings have extremely good oxidation resistance, but suffer from excessive brittleness and fail easily through pinholes. They are useful only for light stress applications where creep cannot occur.

Siliconizing is another protection technique. These coatings are self-healing at high temperatures, since the  $\text{SiO}_2$  layer will creep to cover small imperfections. One commercial organization is producing coatings of this nature first by electroplating a chromium layer onto the molybdenum, then pack siliconizing at high temperature. The process is expensive, but well-adapted to unusual shapes and sizes.

Considerable recent success has been noted by electroplating alternate layers of different metals to make composite coatings which have properties similar to homogenous alloy claddings. For instance, excellent protection has been developed for molybdenum possessing an electroplated layer of chromium under nickel under an outer electroplated chromium layer. This gives the oxidation-resistant behavior of a Nichrome-type metal cladding without the neces-

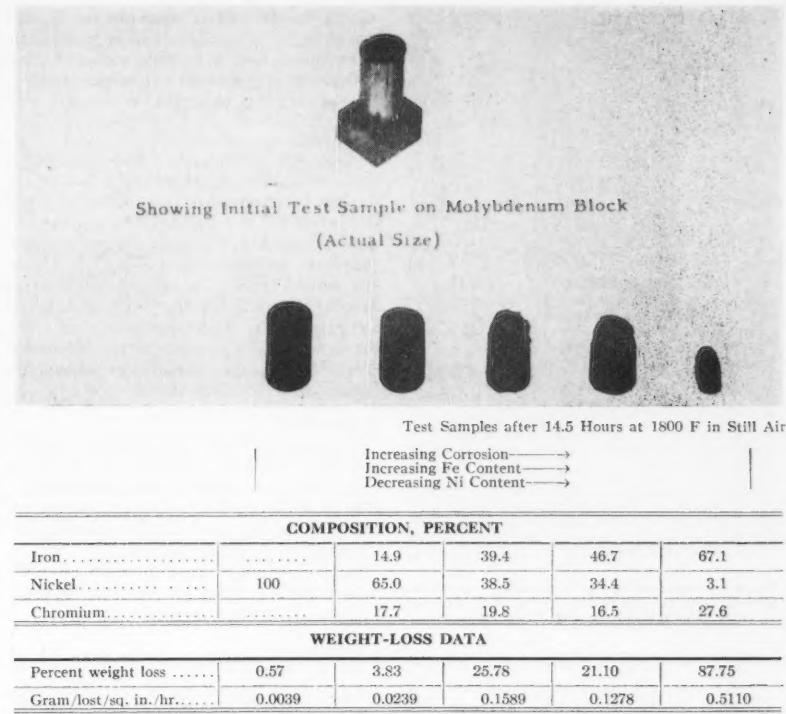


Figure 5—Effects of varying iron and nickel content on the resistance of Fe-Ni-Cr alloys to oxidation in the presence of  $\text{MoO}_3$  at 1800 F.

sity of expensive co-fabrication procedures. Furthermore, it is highly adaptable to irregular shapes.

#### Columbium

Columbium is the second most important refractory metal in terms of technical interest and research dollars. The reasons for columbium's popularity have been well publicized. Unlike molybdenum, it has a relatively refractory oxide,  $\text{Cb}_2\text{O}_5$ , which melts at approximately 1460 C. The appearance of columbium oxidized at 1000 C in air is shown in upper section of Figure 6. The advantages of a refractory metal base where the oxide is not lost by direct volatilization are obvious. At least 15 major research organizations have been investigating the possibilities of further improving the oxidation resistance of columbium by alloying. In addition to its potential as an oxidation-resistant alloy base, columbium has the lowest nuclear cross section for thermal neutrons of the refractory metals. Its density is roughly equivalent to stainless steel. Compared to molybdenum, it has better thermal and electrical conductivity, and because of the greater tolerance of Group V metals for impurities, it is much more workable than molybdenum.

The many searches for columbium-base oxidation-resistant alloys have been fruitful.<sup>11, 12, 13, 14, 15</sup> In a number of cases oxidation resistance approaching stainless steel and Nichrome has been reported. Although some of these alloys do not possess good mechanical charac-

teristics, ideal columbium-base oxidation-resistant alloys with good strength and ductility now appear to be within grasp.

Most of the highly oxidation-resistant columbium-base alloys are based on a few binary alloy systems to which ternary and possibly quarternary alloying additions have been made to achieve further improvement. Only the more interesting binary alloys will be considered here in detail. The Battelle work will be used as an example. Data from organizations such as NASA, Sylvania, Fansteel, Ohio State University and others generally confirm this work.

#### Binary Alloying Elements

The principal binary alloying elements of interest are molybdenum, vanadium, chromium, titanium, and zirconium. Depending on how the binary element affects the oxidation mechanism of columbium, varying amounts are required. The net effect of these additions are shown in Figure 7, and discussed in more detail below:

#### Molybdenum

Molybdenum is the most efficient binary alloying addition, in that optimum protection occurs at approximately 7.5 a/o (atomic percent). It is believed that molybdenum enters substitutional solid solution for columbium in the scale, which is principally  $\text{Cb}_2\text{O}_5$ . Molybdenum has a smaller crystal ion radius than columbium (Table 1) so that the oxide-to-metal volume ratio is decreased to a more favorable value than found for un-

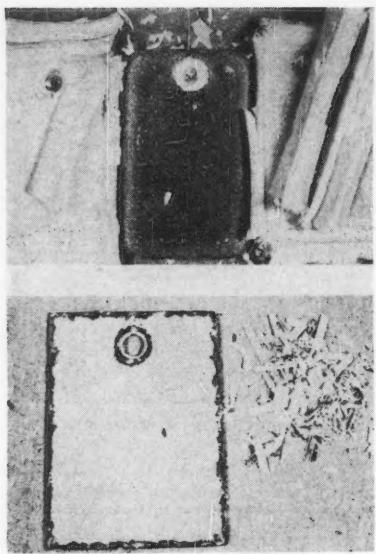


Figure 6—Appearance of unalloyed columbium (above) and of columbium-25 percent titanium alloy after oxidation at 1000°C for six hours.

alloys, except that columbium oxide tolerates more vanadium before solubility is exceeded and a volatile oxide,  $V_2O_5$ , forms. Thus, optimum vanadium content is about 12.5 a/o.

#### Titanium

Titanium creates a different protection mechanism. Considerable binary titanium content is required, since improvement is achieved through formation of double oxides with more mechanical stability than  $Cb_2O_5$ . These are  $Cb_2O_5 \cdot TiO_2$  at about 20-25 a/o titanium and  $3 Cb_2O_5 \cdot TiO_2$  at higher alloy contents. The appearance of oxidized Cb-25 Ti compared to unalloyed columbium is shown in lower section of Figure 6.

#### Zirconium

Columbium base alloys with zirconium are poor at low concentrations, but alloys near 50 a/o zirconium show good oxidation resistance due to formation of a complex oxide scale,  $Cb_2O_5 \cdot 6 ZrO_2$ , analogous to columbium-titanium alloys. The low nuclear cross-section of such alloys makes them of obvious interest for atomic energy applications.

#### Chromium

It is believed that the presence of  $Cr_2O_3$  as a second oxide combines with  $Cb_2O_5$  to form a more refractory oxidation-resistant scale. Mild improvement is achieved.

The net effects of the binary alloying elements on oxidation rate relative to other important high-temperature metals such as molybdenum and stainless steel are shown in Figure 8. Unalloyed columbium is approximately one order of magnitude better than unalloyed molybdenum. Binary alloying elements such as titanium or vanadium produce an additional order of magnitude improvement to about the level of mild steel. Another order of magnitude improvement is needed to attain the level of stainless steels, and still further improvement needed to produce alloys which oxidize as slowly as Nichrome. Promising additions toward these ends are aluminum, silicone and combinations of the basic binary elements. Surface treatments such as aluminizing, siliconizing, and chromizing, common for molybdenum would also appear to have merit and should be investigated.

An additional method of protecting columbium from oxidation is metallic cladding. Loss of columbium through a cladding defect is not probable, as it is in the case of molybdenum, since  $Cb_2O_5$  is not volatile. However, compared to the solid achievements in cladding of molybdenum, progress in cladding columbium has been slow. Columbium forms many compounds and low-melting eutectics with potential cladding metals, so that the stability of clad-to-core bonds is difficult to attain. In general, no successful columbium-base clad materials have been announced.

#### Tantalum

Despite disadvantages of higher density, higher thermal neutron cross-section, and considerably lower availability, tan-

talum metal on first consideration has even more promise than columbium as an oxidation-resistant alloy base. For instance, the principal tantalum oxide melts about 500 degrees above columbium oxide (Table 1).

Columbium and tantalum, sister elements in group V, are similar in many ways. If success achieved in columbium-base oxidation-resistant alloys can be applied to tantalum systems, alloys useful in air up to temperatures of the order of 1400 to 1600°C might be possible. However, initial results of two research efforts show something less than great promise. A report from Fansteel<sup>11</sup> shows only moderate improvement can be obtained by adding up to 30 a/o titanium, 30 a/o iron, 10 a/o cobalt or 10 a/o nickel. Ternaries with carefully chosen binary and ternary additions such as Ta-20 Cr-12 Ni and Ta-20 Cr-12 Co are also mildly promising, for the obvious reason that Ni-Cr or Co-Cr spinels are formed. The Fansteel work also shows that up to 30 a/o molybdenum and vanadium and 40 a/o tungsten were ineffective. In preliminary work by Battelle, these data were generally confirmed; it was also noted that minor improvement could be achieved by addition of small amounts of zirconium or hafnium.

Despite these lackluster data, interest in tantalum metal should increase rapidly until its promise as an oxidation-resistant alloy base is proven or disproven. To date, if attempts to produce tantalum clad with an oxidation-resistant alloy have been made, they have not been published.

#### Tungsten

Tungsten is the most refractory of all metals and thus is the ultimate as an alloy base when high-temperature uses are considered. The properties of its principle oxide  $WO_3$ , place it in an intermediate position among oxidation-resistant alloy bases. Tungsten is a more refractory Group VI metal than molybdenum. Its oxide is also more refractory (See Table 1) and melts at 1470°C. Unfortunately, the vapor pressure of  $WO_3$  is high at temperatures above about 1100°C, so that loss of oxide becomes significant before the melting point is reached; behavior is reminiscent of  $MoO_3$ . In addition, it has been reported that tungsten oxide is seriously affected by moisture to cause an increase in oxidation rate. Fundamental studies of the effects of binary alloying elements on the oxidation resistance of tungsten have not been reported. However, tungsten forms a refractory tungstate with nickel,  $NiWO_4$ , so nickel-base alloy claddings on tungsten should be as interesting as nickel-base claddings on molybdenum.

#### Rhenium

Rhenium metal is the most scarce of the truly refractory high strength metals. Thus, its use is expected to be limited to electronic and electrical applications, where low volume consumption and outstanding properties are characteristic of the materials requirements.

As shown in Figures 1, and 2, rhenium possesses an oxide,  $Re_2O_7$ , which

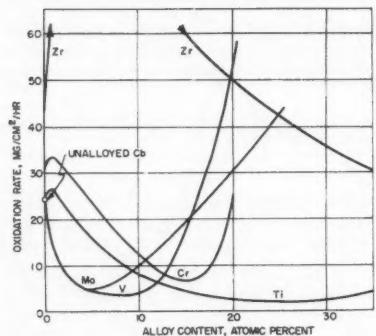


Figure 7—Effect of alloying elements on the oxidation of columbium in air at 1000°C.

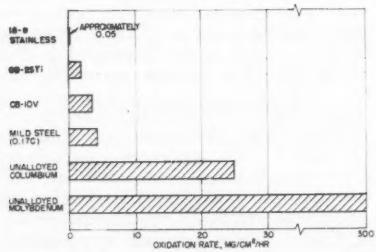


Figure 8—Comparative oxidation rates of high temperature construction materials in air at 1000°C.

alloyed columbium. Molybdenum additions improve oxidation behavior until the solid solubility of molybdenum in the oxide scale is exceeded. At this point molybdenum oxide,  $MoO_3$ , forms and volatilizes causing the catastrophic attack above 7.5 a/o molybdenum shown in Figure 7.

#### Vanadium

The behavior of columbium-vanadium alloys is very similar to the molybdenum

volatilizes at an even lower temperature than molybdenum trioxide, 353 C. It is molten at 297 C. Rhenium is not worth considering at the present state of technology as an oxidation-resistant alloy base although clad rhenium would be feasible.

### Summary

The current state of knowledge of oxidation resistance and protection of the more important refractory metals can be summarized as follows:

Molybdenum is probably the most advanced refractory metal, but it cannot be made oxidation resistant by alloying alone. It must be protected by cladding or coating. Technology has advanced to the point where service of protected molybdenum is imminent in devices such as gas turbine engines. In general, iron-base alloys should be avoided for protecting molybdenum.

Columbium is a very promising, oxidation-resistant alloy base. Study is proceeding at a rapid rate, and strong oxidation-resistant alloys for aircraft, space

vehicle and nuclear applications are a distinct possibility for the moderately near future.

The properties of tantalum and its oxides suggest considerable promise as an oxidation resistant alloy base. Initial results from alloy studies throw doubt on this promise, but research work is proceeding and will continue at an increasing rate until considerably more information is available.

Interest in tungsten is developing, and results of research work can be expected in the near future. Its future in high-temperature technology is assured since it is the most refractory of all metals. However, coating or cladding to produce oxidation resistance is mandatory.

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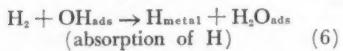
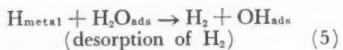
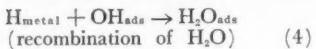
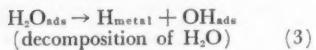
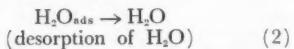
# Kinetics of Absorption by Metals of Hydrogen From Water and Aqueous Solutions\*

By T. G. OWE BERG

## Introduction

THE KINETICS of absorption by metals of hydrogen from  $H_2$  gas was treated in a preceding paper.<sup>1</sup> The same treatment applies with some modification to the absorption of hydrogen from acid solutions and from water. These latter cases will be discussed in this paper.

The general principles will first be reviewed briefly. The basic reactions are:



When the metal is exposed to moist  $H_2$  gas, equilibria are rapidly established between reactions (1) and (2) and between reactions (3) and (4). Desorption is effected by reaction (5); absorption from  $H_2$  gas by reaction (6).

The solution of the system of rate equations corresponding to this system of reaction formulae leads to the following results:

1. Absorption occurs according to a parabolic rate law, the rate being proportional to  $\sqrt{t}$  and also to  $(H_2)^{1/2}$ , when  $t$  is time and  $(H_2)$  the  $H_2$  gas pressure.

2. Desorption occurs according to a hyperbolic or a logarithmic rate law, depending upon the degree of moisture.

3. Absorption and desorption are accelerated by moisture. In particular, desorption in a carefully dried atmosphere is very slow.

4. The solubility is proportional to  $(H_2)^{1/2}$ .

The results 1 and 4 have been confirmed by numerous experimentalists referred to in the preceding paper.<sup>1</sup> The results 2 and 3 have been confirmed by Hudson et al<sup>2-5</sup>, who have studied the effect of moisture extensively. Other observations to the same effect were referred to in the preceding paper.<sup>1</sup>

A conclusion from these results is that the migration of hydrogen in solution in the metal is rapid as compared to the surface reactions for absorption and desorption. This conclusion is directly confirmed by the observation that the rate of desorption is independent of the thickness of the specimen, for steel in the range 3/64 inch to 3/16 inch<sup>4</sup> and for Pd in the range 5 to 20 mils.<sup>6</sup> Similar ob-

servations were referred to in the preceding paper.<sup>1</sup>

It should be emphasized that this treatment pertains to hydrogen in solid solution in the metal (e.g., ferrite), and that the results and conclusions are valid only for this form of hydrogen. In addition, hydrogen may be held as "retained hydrogen." The nature and behavior of retained hydrogen in steel will be discussed elsewhere. It may be mentioned, however, that it occurs in a precipitate,  $\epsilon$ -carbide, of the composition  $Fe_2HC'$ . It will be assumed in the following that no retained hydrogen is present.

The agreement, just referred to, between the reaction mechanism (1)-(6) and the experimental data may be checked further by application to related processes. Such checking will be done in the following by application to hydrogen absorption from dilute acid solutions and from water and thereby to the corrosion of metals in these agents.

## Basic Rate Formulae

As in the preceding paper,<sup>1</sup> concentrations will be denoted by parenthesis and brackets, parenthesis for species in the gaseous or liquid environment, square brackets for coverages on the metal surface, and wavy brackets for concentrations in the metal.

The equilibria between (1) and (2) and between (3) and (4) are expressed by the formulae

$$(H_2O) (1 - \Theta) = K [H_2O] \quad (7)$$

$$\{H\} [OH] = K'' [H_2O] \quad (8)$$

where  $\Theta$  is the fraction of the surface area covered by  $OH_{ads}$  and  $H_2O_{ads}$ , i.e.,

$$\Theta = [OH] + [H_2O] \quad (9)$$

and  $K$  and  $K''$  are equilibrium constants. Of these,  $K''$  is associated with zero activation energy, whereas  $K$  is associated with an activation energy equal to the difference between that for desorption and that for adsorption of  $H_2O$ .

The solutions of equations (7), (8), and (9) are

$$\frac{(H_2O)}{[H_2O]} = \frac{(K'' + \{H\}) (H_2O) + K (H)}{(K'' + (H)) (H_2O) + K (H)} \quad (10)$$

$$\frac{[OH]}{[H_2O]} = \frac{K'' (H_2O)}{(K'' + (H)) (H_2O) + K (H)} \quad (11)$$

$$1 - \Theta = \frac{K (H)}{(K'' + (H)) (H_2O) + K (H)} \quad (12)$$

In the particular case that  $(H_2O) \gg K$ ,  $\{H\} \gg K''$ , these formulae reduce to

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## Abstract

The kinetics of absorption of hydrogen in corrosion is treated on the basis of a mechanism previously proposed for the absorption of hydrogen from  $H_2$  gas. Rate formulae are derived and compared with experimental data. On certain simple assumptions, a parabolic or a cubic rate law is derived for an initial period, during which absorption of hydrogen occurs. This rate law is succeeded by a linear rate law when the metal is saturated with hydrogen. The rate in acid solutions is proportional to the square root of the cube root of the acid concentration. The rate in water vapor is proportional to the square root of the water vapor pressure or independent of the water vapor pressure. 3.84

It should not require rate constants with zero activation energy.

The variation of rate for two cases:

$$[H_2O] = \sim 1 \quad (13)$$

$$[OH] = \sim \frac{K''}{\{H\}} \quad (14)$$

$$1 - \Theta = \sim \frac{K}{(H_2O)} \quad (15)$$

The rate of absorption from  $H_2$  gas is

$$\frac{d(H)}{dt} = \frac{2k_1}{d} (H_2) [OH] (1 - \Theta) = \frac{2k_1}{d} KK'' \frac{(H_2)}{(H_2O)} \frac{1}{\{H\}} \quad (16)$$

when  $d$  denotes the thickness of the sheet specimen.

Integration gives<sup>1</sup>

$$\{H\} = \sqrt{\frac{2k_1}{d} KK''} \frac{(H_2)^{1/2}}{(H_2O)^{1/2}} \sqrt{t} \quad (17)$$

The rate of desorption is<sup>1</sup>

$$\frac{d(H)}{dt} = -k_2 (H) [H_2O] \quad (18)$$

Equating the rates (16) and (18) gives for the solubility<sup>1</sup>

$$\{H\} = \sqrt{\frac{2k_1}{k_2} \frac{KK''}{d} \frac{(H_2)^{1/2}}{(H_2O)^{1/2}}} \quad (19)$$

## Dissolution of Hydrogen-Replacing Metals in Dilute Acids

When a hydrogen-replacing metal is immersed in a dilute acid solution, the primary reaction is one between metal

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and water, whereas the acid reacts indirectly by dissolving the hydroxide formed in the primary reaction. There are several reasons for this situation:

1. The acid concentration is much smaller than that of the water.

2. The reaction products of metal and acid dissolve rapidly (i.e., the acid is rapidly desorbed, whereas adsorbed water is slowly desorbed and the reaction product of water, hydroxide, is extremely slowly desorbed, except by reaction with the acid.)

3. The activation energy for the adsorption of acid is equal to the strength of the bonds by which the acid is held in the solution (i.e., the heat of vaporization at  $\theta$  K of the acid in solution<sup>a, b</sup>). This activation energy is very high as compared to that for the adsorption of water.

Thus, the rate of adsorption of acid,  $A_c$ ,

$$\frac{d[A_c]}{dt} = k_a [OH](Ac) \quad (20)$$

is very small.

The rate of dissolution of the hydroxide may be assumed to be proportional to  $[OH]$  under certain conditions. Thus,

$$v = k_s [OH](Ac) \quad (21)$$

It should be noted that this process does not require the adsorption of acid. The rate constant  $k_s$  is therefore associated with zero activation energy. Furthermore, the factor  $(1 - \Theta)$  does not occur in the rate formula (21).

The value of  $[OH]$  to be used in the rate formula (21) will be calculated for two cases.

1. The dissolution in a sufficiently dilute acid is too slow to materially affect the equilibria (7) and (8). Hence,  $[OH]$  may be taken from the formula (14). The dissolution is accompanied by evolution of  $H_2$  gas, the pressure of which is equal to the atmospheric pressure plus the hydrostatic head. Formula (17) may be used for  $\{H\}$ , inserting  $\{H_2\} = 1$ . This gives

$$v = k_s \sqrt{\frac{d}{2k_s K''}} \frac{(H_2O)^{\frac{1}{2}}}{\sqrt{t}} (Ac) \quad (22)$$

Thus, the rate is proportional to the acid concentration, and the dissolution follows a parabolic rate law.

2. At a stationary state, the rate of dissolution of  $OH_{ads}$  is equal to the rate of formation of  $OH_{ads}$ . When these rates are high, the equilibrium formula (7) does not hold. However, the formula (8) is still valid. At this stationary state,

$$k_s [OH](Ac) = k_t [H_2O] (1 - \Theta) \quad (23)$$

This may also be expressed in terms of  $(1 - \Theta)$ , the value of  $(1 - \Theta)$  being constant at the stationary state. Thus,

$$\frac{d(1 - \Theta)}{dt} = k_s [OH](Ac) - k_t [H_2O] (1 - \Theta) = 0 \quad (24)$$

This gives

$$1 - \Theta = \frac{k_s}{k_t} \frac{[OH](Ac)}{[H_2O]} \quad (25)$$

and, with  $[H_2O]$  and  $[OH]$  from formulae (13) and (14),

$$1 - \Theta = \frac{k_s}{k_t} K'' \frac{(Ac)}{\{H\}} \quad (26)$$

The rate of formation of  $H_{metal}$  is equal to the rate of formation of  $OH_{ads}$ . Thus,

$$v = d \frac{d\{H\}}{dt} = k_t [H_2O] (1 - \Theta) \quad (27)$$

$$k_s K'' \frac{(Ac)}{\{H\}} \quad (27)$$

Integration gives

$$\{H\} = \sqrt{\frac{2k_s}{d} K''} (Ac)^{\frac{1}{2}} \sqrt{t} \quad (28)$$

This inserted in the rate formula (21) or (27) gives

$$v = d \frac{d\{H\}}{dt} = \sqrt{\frac{d}{2} k_s K''} \frac{(Ac)^{\frac{1}{2}}}{\sqrt{t}} \quad (29)$$

In this case, the rate is proportional to the square root of the acid concentration. The dissolution follows a parabolic rate law.

A variation of this case is when the rate is proportional to  $[OH]^2$  (i.e., when  $OH_{ads}$  is not formed in pairs). This corresponds to a still higher rate of dissolution with low values of  $[OH]$ . The rate formula is now, instead of formula (21),

$$v = k_s [OH]^2 (Ac) \quad (30)$$

This gives, instead of formula (27),

$$v = d \frac{d\{H\}}{dt} = k_s K''^2 \frac{(Ac)}{\{H\}^2} \quad (31)$$

Integration gives

$$\{H\} = \left( \frac{3}{d} k_s K''^2 \right)^{\frac{1}{3}} t^{\frac{1}{3}} \quad (32)$$

and

$$v = \frac{1}{3} (3d^2 k_s K''^2)^{\frac{1}{3}} (Ac)^{\frac{1}{3}} t^{-\frac{2}{3}} \quad (33)$$

The rate is now proportional to the cube root of the acid concentration, and the dissolution follows a cubic rate law. At saturation of the metal with hydrogen, the rates of absorption and desorption are equal. This condition gives a value of  $\{H\}$  to be inserted in the formula (14) for  $[OH]$  and in the rate formulae. Taking  $\{H\}$  from formula (19) gives (case 1)

$$v = k_s \sqrt{\frac{k_s d K''}{2 k_t K}} (H_2O)(Ac) \quad (34)$$

Equating the rates (18) and (27) gives (case 2)

$$\{H\} = \sqrt{\frac{k_s K''}{k_t d}} (Ac)^{\frac{1}{2}} \quad (35)$$

This inserted in the rate formula (27) gives

$$v = \sqrt{k_s k_t K'' d} (Ac)^{\frac{1}{2}} \quad (36)$$

Equating the rates (18) and (31) gives (case 2)

$$\{H\} = \left( \frac{k_s K''^2}{k_t d} \right)^{\frac{1}{3}} (Ac)^{\frac{1}{3}} \quad (37)$$

This inserted in the rate formula (31) gives

$$v = (k_s K'' d)^{\frac{1}{3}} (Ac)^{\frac{1}{3}} \quad (38)$$

Thus, when the metal is saturated with hydrogen, the metal dissolves at a constant rate that is proportional to the same power of the acid concentration as is that in the initial parabolic or cubic state of dissolution.

### Reaction of Hydrogen-Replacing Metals With Water Vapor

The essential difference between the reactions of hydrogen-replacing metals with acid solutions and with liquid or gaseous water is that the acid dissolves the hydroxide formed, whereas the water does not. In the latter case,  $OH_{ads}$  must be removed from the metal surface by some other process (e.g., by diffusion into a coating of oxide or hydroxide). The rate of this process may be assumed to be proportional to  $[OH]$  or to  $[OH]^2$ . In the former case

$$v = k_s [OH] \quad (39)$$

in the latter case

$$v = k_t [OH]^2 \quad (40)$$

The solutions from the preceding section may be taken over by replacing  $k_s (Ac)$  by  $k_s$  and  $k_t (Ac)$  by  $k_t$ . This gives

$$v = k_s \sqrt{\frac{d}{2k_s K''}} \frac{(H_2O)^{\frac{1}{2}}}{\sqrt{t}} \quad (41)$$

$$v = \sqrt{\frac{d}{2} k_s K''} \frac{1}{\sqrt{t}} \quad (42)$$

$$v = \frac{1}{3} (3d^2 k_s K''^2)^{\frac{1}{3}} t^{-\frac{2}{3}} \quad (43)$$

### Comparison With Experimental Data

#### Dissolution of Hydrogen-Replacing Metals in Dilute Acids

The type of rate law encountered in the dissolution of a hydrogen-replacing metal in a dilute acid depends very much upon the conditions at hand. A parabolic rate law is typical of the case when the removal of hydrogen from the surface, notably by absorption, is rate-determining. A parabolic rate law has been found with  $Fe^{10}$ ,  $Al^{10}$ , and  $Ni^{10}$  in  $HCl$  solutions, with  $Ni^{11}$ ,  $Fe^{10}$  and steel<sup>10</sup> in  $H_2SO_4$  solutions, and with  $Ni^{11}$  in  $HNO_3$  solutions. A cubic rate law has not occurred in the experience of the author.

A parabolic or a cubic rate law shows that the products oppose the reaction according to the formula

$$\frac{dx}{dt} = \frac{k}{x} \quad (44)$$

or

$$\frac{dx}{dt} = \frac{k}{x^2} \quad (45)$$

A variety of mechanisms may conceivably lead to such rate formulae. A better check on the mechanism is offered

therefore by the reaction order with respect to the acid concentration. Furthermore, the parabolic rate constant can rarely be determined with an accuracy comparable to that of a linear rate constant. Parabolic curves are frequently erratic, switching from one rate law to another in the course of the experiment. A peculiar phenomenon, observed particularly clearly with pure Fe (Armco-iron) and pure Al (99.9 percent) in HCl solutions,<sup>8</sup> is the stepped parabolic curve consisting of a sequence of parabolae. This phenomenon seems to be typical of extremely slow desorption of hydrogen. It appears to be accompanied by blistering of the metal, although such may not always be the case.

For these reasons it is preferable to check the formulae derived by comparison with the linear branch of the dissolution curve that follows upon the initial parabolic or cubic curve. Rates proportional to the square root of the acid concentration have been observed with Ni<sup>II</sup> and Fe-Cr alloys<sup>12</sup> in HNO<sub>3</sub> solutions and with Ni<sup>II</sup> and Armco-iron<sup>10</sup> in HCl solutions. In these cases, the dissolution followed an initially parabolic rate law. Rates proportional to the cube root of the concentration have been observed with Ni<sup>II</sup> and Armco-iron<sup>10</sup> in H<sub>2</sub>SO<sub>4</sub> solutions and with Al in NaOH solutions.<sup>13</sup> A square root dependence also has been observed with Al in NaOH solution.<sup>14</sup>

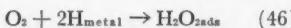
#### Reaction of Hydrogen-Replacing Metals With Water Vapor

Among the three rate formulae derived for this process, two are parabolic and one cubic; one, (41), depends on the H<sub>2</sub>O vapor pressure whereas the other two, (42) and (43), are independent of the H<sub>2</sub>O vapor pressure.

Numerous cases of parabolic and cubic rate laws for the oxidation of metals are cited by Kubaschewski and Hopkins.<sup>15</sup> These have not always been associated with water vapor. On the other hand, it is difficult to conceive of experimental conditions for oxidation of metals which would completely exclude the presence of water. An accelerating effect of water upon the oxidation of metals in an oxygen atmosphere has been observed with Al,<sup>16-19</sup> Zn,<sup>20</sup> Mg,<sup>21-24</sup> Cu,<sup>25</sup> Be,<sup>26</sup> Nb,<sup>27</sup> Fe,<sup>28</sup> low-carbon steel,<sup>29</sup> and Fe alloys.<sup>30-32</sup> In some cases, the process came to a complete stop in the absence of moisture,<sup>33, 34</sup> presumably as the initial water was used up. Steel does not rust in a dry climate.<sup>35</sup>

Although water is instrumental in the mechanism under consideration, it is only at the lowest rates (i.e., at the lowest levels of moisture), that the water vapor pressure enters into the rate formula. Hence at a high moisture content, in one case<sup>36</sup> above 4 percent, addition of more water vapor does not affect the rate. The rate of oxidation of Mg in moist O<sub>2</sub> is proportional to (H<sub>2</sub>O)<sup>1/2</sup> in agreement with the rate formula (41).<sup>24</sup>

The rate of oxidation in an oxygen atmosphere is frequently independent of or depends erratically on the O<sub>2</sub> pressure.<sup>15</sup> Independence of O<sub>2</sub> pressure shows that O<sub>2</sub> does not take part in the rate-determining step. The major role of O<sub>2</sub> is to react with adsorbed hydrogen,



or



The reaction (47) increases the rate of oxidation by removing H<sub>metal</sub> and by forming OH<sub>ads</sub> and H<sub>2</sub>O<sub>ads</sub> (reactions (47) and (4)).

The stepped parabolic curve also has been observed in oxidation, particularly in moist atmospheres.<sup>15, 30, 34, 35</sup> Heindlhofer and Larsen<sup>36</sup> and others<sup>37</sup> have associated this phenomenon with blistering of the oxide layer. The author<sup>10</sup> has found such stepped curves in the oxidation of iron that contained internal voids, formed in the fusion of the iron; the phenomenon was enhanced by moisture.

This phenomenon cannot be explained at this time. The rate changes very suddenly as a saturation level is approached. This is typical of hydrogen absorption. The amount of hydrogen absorbed plotted against  $\sqrt{t}$  follows a perfectly straight line and then suddenly switches over to a constant level at saturation.<sup>2, 4, 37</sup> The net rate of absorption thus follows a hyperbola when plotted against  $\sqrt{t}$  and suddenly drops to zero at saturation. This shows that there is no appreciable desorption during absorption, for otherwise saturation would be approached asymptotically. It follows that the rate of desorption is essentially zero until saturation, when it suddenly jumps to a value equal to the rate of absorption. In the case of a stepped parabolic curve, the rate of desorption suddenly jumps to a value higher than the rate of absorption and then as suddenly falls back to zero. Thus, the stepped parabolic curve is caused by sudden bursts of hydrogen desorption. The mechanism by which these discontinuities are caused cannot be discussed until additional information becomes available. However, it may be concluded from the occurrence of a stepped parabolic curve that hydrogen absorption and desorption are involved in the process.

#### Conclusion

It has been assumed in this paper as well as in the preceding paper<sup>1</sup> that the metal surface is covered by OH<sub>ads</sub> and H<sub>2</sub>O<sub>ads</sub> and that no other adsorbed species of variable coverages are present. This assumption holds in some cases, in other cases it does not. In some cases, the metal surface is covered by other reaction products, such as oxide or sulfates, to an extent that varies with time and other parameters. These additional variable coverages must be taken into account in the derivation of the rate law. This applies particularly to the absorption and desorption of hydrogen in a dried atmosphere or in a vacuum. In these instances, traces of surface oxide may be the major source of the OH<sub>ads</sub> and H<sub>2</sub>O<sub>ads</sub> required. This more general case has been treated elsewhere.<sup>38</sup>

The rate formulae derived in this paper and the preceding paper are limited in their applicability accordingly. Nevertheless, they are in agreement with empirical rate formulae. In view of the limitation just referred to, this agreement does not constitute final proof of the correctness

of the mechanism under consideration. The occurrence of the square root or cube root of the concentration in the rate formula for the dissolution of metals may also be caused by equilibria established at or on the metal surface.<sup>39, 40</sup> However, such equilibria are not likely to occur in the cases under consideration.

It may be concluded that the mechanism under consideration is compatible with experimental data and observations in cases where absorption of hydrogen is part of the process. This lends additional support to this mechanism and to the conclusion in the preceding paper<sup>1</sup> that migration of hydrogen in solution in the metal is rapid as compared to the surface reactions for absorption and desorption of hydrogen.

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# Corrosion Inhibitor Evaluation At Elevated Temperatures\*

By VICTOR W. MAXWELL

## Introduction

MANY DIFFERENT types of evaluation tests have been devised for screening down-the-hole oil and gas well corrosion inhibitors. The tests vary in type from sour crude to sweet crude, static<sup>1</sup> to dynamic,<sup>2, 3</sup> room temperature to elevated temperatures,<sup>3</sup> small rod type coupons to larger area coupons, weight loss determination direct or by weight loss determinations by electrical resistance,<sup>4</sup> and visual comparison to statistical analyses of many numerical results.<sup>5</sup> One method involves the use of electronic bridge circuits to measure the adsorbed film resistance.<sup>6</sup> The common goal is, of course, to find suitable inhibitors for field use from the many commercially available inhibitors on the market today. Considering the variables involved in such an undertaking, it is by no means a simple task to arrive at one single test which will yield concrete and reproducible answers that are indicative of the quality of the inhibitor in question.

At best, the results of any laboratory evaluation test give only an indication as to the ability of an inhibitor to protect against down-the-hole corrosion. Therefore, the results from any one evaluation test should not be taken conclusively; rather, the results should narrow the field to a few inhibitors to be screened by other laboratory and field tests for possible use in the field.

The choice of the evaluation test to be used should be based on such items as: (1) time required to run the test, (2) availability of materials, (3) equipment necessary for running the test, (4) simplicity in method and interpretation, (5) reproducibility of results, and (6) close approximation to actual field conditions. The last item is perhaps the most important but needs to be qualified further. Since it is impossible to repro-

duce exact field conditions in the laboratory, steps should be taken to simulate closely these conditions by controlling the established variables which have a decided effect on corrosion rates in oil and gas wells. Some of these variables are: (1) the composition and percent by volume of brine used, (2) the type and percent by volume of the oil phase used, (3) the type and concentration of gas phase used, and (4) temperature. The composition of the brine should be representative of actual field brines where the compounds which are known to contribute to down-the-hole corrosion are concerned (i.e., salt content, soluble organic acid content, and dissolved gas content).

One variable which recently has been recognized as important in laboratory evaluation is temperature. The corrosion reaction in question, like most chemical reactions, increases in rate with increase in temperature. By increasing the temperature at which an evaluation test is run, the corrosion rate and the amount of corrosion taking place is subsequently higher for the same period of time. Therefore, laboratory tests which require long periods of time at room temperature to obtain any sizable amount of corrosion are greatly accelerated by an increase in temperature. Also the ability of the inhibitor to protect this increase in amount of corrosion due to an increase in temperature constitutes a better evaluation than the protection by the same inhibitor of a small amount of corrosion at room temperature.

It is the object of this paper to present results of an evaluation of several different inhibitors at elevated temperature as a possible means of classifying the many commercial inhibitors according to their value as down-the-hole inhibitors. All of the inhibitors reported were evaluated in a sweet crude system. The system could be altered to sour crude by using hydrogen sulfide instead of carbon dioxide as the dissolved gas.

## Abstract

Evaluation of commercially available, organic corrosion inhibitors at elevated temperatures is presented. All of these inhibitors were designed for down-the-hole treatment of producing oil and gas wells. The evaluations at elevated temperatures were compared to the evaluations of the same inhibitors run at room temperature. All phases of the test used are discussed, including apparatus, materials used, and test method. A table of results is given, as well as a block graph showing the average percent protection obtained for each inhibitor at the temperatures tested.

23.4

## About the Author



VICTOR W. MAXWELL is in the production laboratory of Sun Oil Company, Gulf Coast Division, in Beaumont, Texas. He is currently engaged in work dealing with the problems of oil and gas condensate well corrosion. Prior to joining Sun Oil Company in February 1954, he was employed by Eli Lilly and Company, Indianapolis, Indiana, as a research chemist in the analytical department of the antibiotics division. He attended Baylor University, Waco, Texas, and received BS and MS degrees in chemistry in 1950 and 1951 respectively. He is a member of NACE.

## Apparatus and Materials

### Apparatus

**The Constant Temperature Box.** The constant temperature box used for this test, shown in Figures 1 and 2, was designed and constructed in this laboratory. It is similar to those used by many other laboratories interested in evaluation of inhibitors.

**Corrosion Cell.** A 250 ml borosilicate nursing bottle with screw cap and neoprene gasket has been used with success in withstanding the leaks and breakages at higher temperatures. A close-up of two of these bottles clamped in the holders and containing the fluids and coupon is shown in Figure 3.

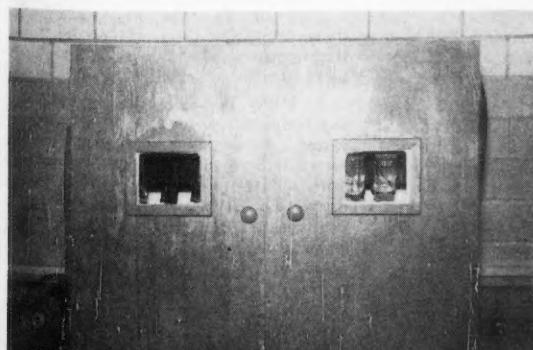


Figure 1—Constant temperature box.

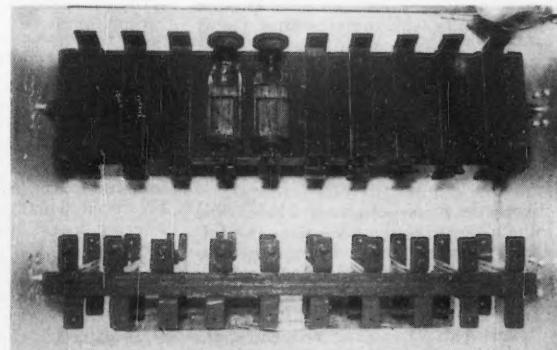


Figure 2—Bottles clamped in holders for constant temperature box.

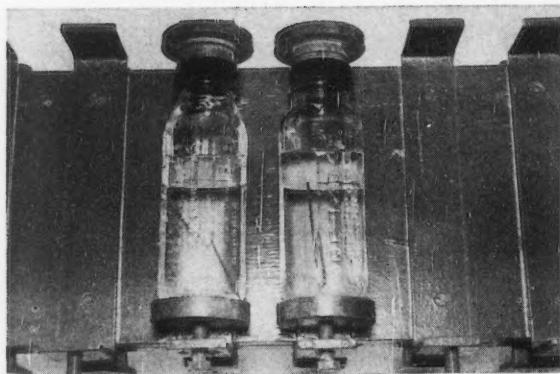


Figure 3—Close-up of bottles clamped in holders.

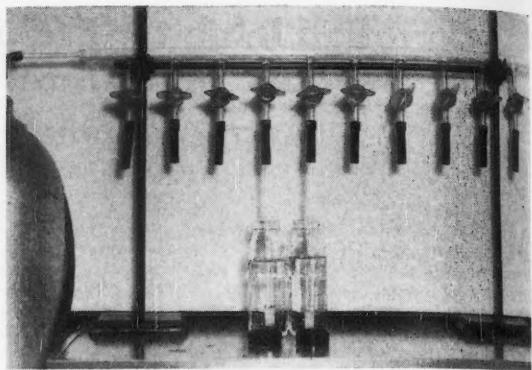


Figure 4—Glass dispersion tubes for saturating fluid with carbon dioxide.

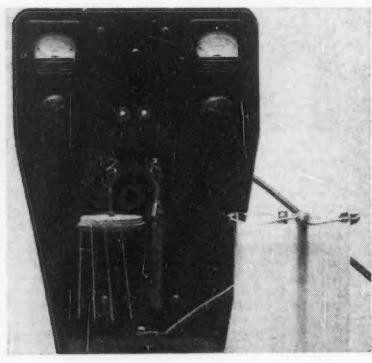


Figure 5—Apparatus for electrolytic cleaning of coupons.

**Coupons.** Drill rods, 1/16" x 4', made of low carbon SAE 1010 steel, were cut in average lengths of 3 1/2 inches. These coupons were cleaned by rotating inside a bottle containing Ottawa sand and benzene. After cleaning, specimens were dried and put in a desiccator until they were weighed and used.

**Apparatus for Saturating Fluid with CO<sub>2</sub>.** A borosilicate manifold arrangement was used to saturate ten cells with CO<sub>2</sub> at a time. This manifold was fashioned from glass tubing and stop-cocks. Medium sintered glass dispersion tubes were used to saturate the fluid. These are removable for cleaning. This apparatus is shown in Figure 4.

**Electrolytic Cleaning Apparatus.** Coupons taken from a test run were cleaned electrolytically with the apparatus shown in Figure 5. This apparatus was built in this laboratory. It is designed to clean either two large field coupons or ten small drill rod type coupons at one time. The two types of holders are shown in Figure 5.

#### Materials

**Synthetic Brine—Aqueous Phase.** The synthetic brine was made up in the following manner. Fifteen liters were prepared at one time by dissolving the weights in grams per fifteen liters given in Table I and diluting to fifteen liters with distilled water. The concentrations of the salts given are based on analyses for these specific salts in field brines and

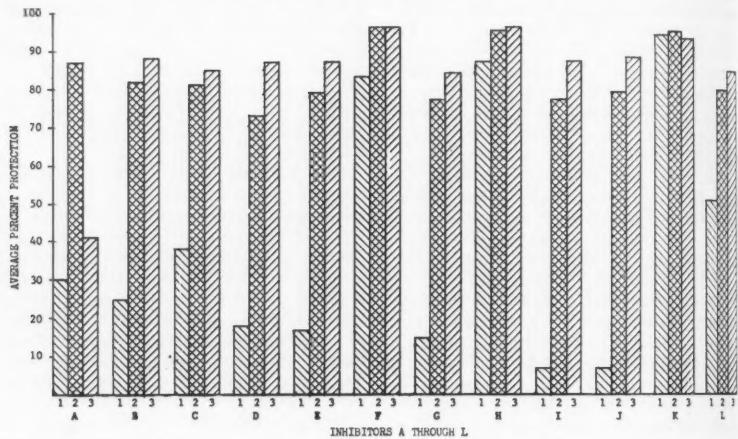


Figure 6—Results of inhibitor evaluation at elevated temperature. Code to numbers is as follows: 1—room temperature; 2—150 F; 3—220 F.

TABLE I—Concentration of Salts

Constituent	gm/liter	gm/15 liter	ppm
Calcium Chloride, CaCl <sub>2</sub> .2H <sub>2</sub> O	13.24	198.6	10,000
Sodium Sulfate, Na <sub>2</sub> SO <sub>4</sub>	3.50	52.5	3,500
Sodium Chloride, NaCl	60.00	900.0	60,000
Magnesium Chloride, MgCl <sub>2</sub> .6H <sub>2</sub> O	10.68	160.2	5,000
Sodium Bicarbonate, NaHCO <sub>3</sub>	1.00	15.0	1,000

constitute a rough average of the concentrations found by this laboratory.

**Hydrocarbon Phase.** Standard grade kerosene, which had been depolarized by agitation with diatomaceous earth and activated charcoal, was used as the hydrocarbon phase.

#### Method

1. One-hundred and sixty ml of brine and forty ml of kerosene were placed in each bottle followed by 0.2 ml of glacial acetic acid (1000 ppm based on total fluid). The appropriate amount of each inhibitor to be tested was placed in all of the designated bottles except those referred to as "blanks".

2. A gas dispersion tube was placed in the fluid, and carbon dioxide bubbled through the mixture for five minutes at room temperature. The weighed coupons were placed in the bottles while the mixture was being saturated with CO<sub>2</sub>. At the end of five minutes, the bottles were

capped and placed in the holders inside the constant temperature box.

3. After the bottles were mounted on boards with steel clamps inside the constant temperature box (see Figure 3), the motor which rotates the boards at a rate of 30 rpm was started. The thermostat was then set for the desired temperature. The bottles were allowed to rotate for a period of 24 hours.

4. At the end of the agitation period, the coupons were removed, cleaned with benzene to remove oil, placed in the electrolytic cleaning bath and descaled with a current of 3 amps for 3 minutes, dipped in 10 percent sodium carbonate to neutralize the 5 percent inhibited sulfuric acid, wiped clean with a towel, and dried by dipping in acetone. They were either weighed immediately or placed in a desiccator for future weighing.

5. The inhibitors tested were evaluated at three different temperatures: namely,

Inhibitor  
Blank...  
A...  
B...  
C...  
D...  
E...  
F...

Blank...  
G...  
H...  
I...  
J...  
K...  
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TABLE 2—Results of Inhibitor Evaluation at Elevated Temperature

Inhibitor	Room Temperature			Run No.	150 F			Run No.	220 F		
	Wt. Loss, mg.	Avg. Wt. Loss-mg.	Avg. % Prot.		Wt. Loss, mg.	Avg. Wt. Loss-mg.	Avg. % Prot.		Wt. Loss, mg.	Avg. Wt. Loss-mg.	Avg. % Prot.
Blank.....	1	13-12-16	14	2	73-107-70	83	87%	3	138	138	41%
A.....	1	9-10-11	10	2	5-16-11	11	87%	3	42-97-105	81	88%
B.....	1	9-11-12	11	2	17-14-14	15	82%	3	18-15-16	17	85%
C.....	1	9-8	9	2	21-14-12	16	81%	3	24-20-18	21	87%
D.....	1	14-11-9	11	2	23-21-25	23	73%	3	14-21-21	18	87%
E.....	1	13-11-11	12	2	18-16-18	17	79%	3	18-21-16	19	87%
F.....	1	4-2-2	3	2	2-4-4	3	96%	3	12-2-3	6	96%
Blank.....	4	20-14-13	16	5	122-42-68	77	77%	6	140-135	138	84%
G.....	4	14-13-13	13	5	23-17-14	18	77%	6	27-18-20	22	96%
H.....	4	1-1-4	2	5	3-6-2	4	95%	6	4-6-6	5	87%
I.....	4	16-17-14	15	5	17-18-18	17	77%	6	21-19-16	19	88%
J.....	4	16-10-16	14	5	16-17-15	16	79%	6	17-15	16	93%
K.....	4	1-1-1	1	5	4-4-3	4	95%	6	8-9-11	9	85%
L.....	4	8-7-8	8	5	16-17-14	16	80%	6	20-24-17	20	85%

(1) room temperature, (2) 150 F, and (3) 220 F. All inhibitors were added at a concentration of 100 parts per million based on total fluid. All blanks and inhibitor samples were run in triplicate. Calculations are based on an average of the three weight losses for each blank and each inhibitor tested.

### Results and Discussion

#### Calculations

The weight losses obtained on the inhibited coupons and the blank coupons are used to calculate the percent protection of the inhibitor being tested. The calculations are simple and are made using the formula given below:

$$\frac{\text{Blank wt. loss} - \text{Inhibited wt. loss}}{\text{Blank wt. loss}} \times 100 = \% \text{ protection}$$

#### Results

Table 2 shows the results of the evaluation of 12 different inhibitors which are established, commercially-available compounds currently in use by this company, as well as by other producing companies, for corrosion treatment of both sweet crude oil wells and gas condensate wells. The table shows the actual weight loss for each sample run and the average weight loss of the triplicate. The average percent protection is calculated from the average weight loss by the formula given previously.

The block graph shown in Figure 6 gives a comparison of the average percent protection for each inhibitor at the three different temperatures. Of the inhibitors tested, F, H, and K were the only ones which exhibited better than eighty percent protection for all three temperatures. Only inhibitor A showed a poor percent protection at 220 F. All of the inhibitors showed better than seventy percent protection at 150 F.

From the preceding statements, the importance of temperature in the evaluation of a group of inhibitors is somewhat lessened since the results indicate that inhibitors F, H, and K are the best inhibitors of the ones tested, regardless of the temperature at which the test was run. However, it is felt that evaluation at the elevated temperatures is more

indicative of the quality of the inhibitor since (1) the percent error in cleaning and weighing of the coupons is not magnified as it is with the small weight losses at room temperature, and (2) the elevated temperature more closely approaches actual well conditions.

Since there is little difference in the results at 150 F and 220 F and since it is easier to run the evaluation at 150 F, this latter temperature has been established as the temperature for further screening of inhibitors in this laboratory on both synthetic and actual sweet crude systems.

#### Conclusions

While it is recognized that this method at elevated temperature is not the final word in inhibitor screening, it is believed to be a good test which is both practical and economical and will help decide upon a few good inhibitors from the many which are available. The results should not be taken conclusively, but should be used jointly to evaluate objectively the inhibitors designed for down-the-hole treatment of sweet gas and oil wells.

#### Acknowledgments

The author acknowledges the efforts of Fritz R. Brandes in completing the experimental work necessary for the writing of this report. Acknowledgments are made to H. W. Perkins and W. F. Oxford, Jr. for their objective and helpful comments concerning the composition and contents of this report. Appreciation is expressed to Sun Oil Company for permission to give this paper.

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### DISCUSSION

#### Questions by J. D. Sudbury, Continental Oil Co., Ponca City, Oklahoma:

- What type chemical structure is most effective at high temperature?
- Do you plan any work at higher temperature?
- Do you plan any work with pure compounds?
- What is the explanation for a maximum protection at 150 F in some inhibitors?

#### Replies by Victor W. Maxwell:

- Since we were not concerned with different types of chemical or molecular structure, I am unable to say which type is most effective at high temperature.
- At present, we do not plan on any work at higher temperature.
- We do not plan any work with pure compounds, inasmuch as we are interested primarily in screening samples of commercially available inhibitors.
- With the exception of Inhibitor A, all of the inhibitors tested showed better percent protection at 150 F and 220 F. I have no explanation for this except that the blanks are greater at higher temperatures, which would diminish the effects of cleaning and weighing errors.

#### Question by G. A. Marsh, The Pure Oil Company, Crystal Lake, Illinois:

What is the effect of time on the performance of inhibitors in this type of test?

#### Reply by Victor W. Maxwell:

In an effort to establish the shortest length of time for running this test, blank samples were run in duplicate at various intervals of time. It was found that the curve (from a plot of time versus weight loss) began to level off at 20 to 24 hours. This leveling probably corresponds with the equilibrium established between corrodents and corrosion products; therefore, running the test for a longer period than 24 hours is somewhat useless.

#### Comments by Jack P. Stanton, Houston, Texas:

I would suggest that the reason that most of the inhibitors looked good at 150 F was simply that most of the sup-

pliers did the bulk of their screening of sweet inhibitors in similar fluids at approximately the same (150 F) temperature.

**Question by Ben F. Davis, Houston, Texas:**

Do you suppose the reason for greater effectiveness of most of the inhibitors at higher temperatures is due to the lower carbonic acid concentration in the liquid phase?

**Reply by Victor W. Maxwell:**

Perhaps so, but consider the possibility that as the corrosion reaction pro-

ceeds, the concentration of carbonic acid in the liquid phase decreases; therefore, more carbon dioxide from the gas phase is dissolved. This would constitute a more aggravated attack at higher temperature than at room temperature.

**Question by William P. McKinnell, Jr., Ohio Oil Co., Littleton, Colorado:**

Do you feel that the complex composition of the brine used in these tests improves the reliability of the tests? Have any tests been duplicated using a straight 5 percent NaCl brine in place of the complex brine? There is always the chance of human error in preparing such solutions. Small composition changes in

complex brines can result in large changes in corrosion rate. In corrosion testing therefore, it is usually wise to use the simplest environment which gives reliable results.

**Reply by Victor W. Maxwell:**

Only to the extent that the synthetic brine used in this test more closely simulates actual field brines than does a straight 5 percent sodium chloride brine. We have not duplicated these tests using a straight 5 percent sodium chloride brine in place of the complex synthetic brine used. Human error can occur with the preparation of any solution, simple or complex.

**Any discussion of this article not published above  
will appear in the December, 1960 issue.**

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# The Corrosion Behavior of Welded Low Alloy Steel\*

By D. C. BRIGGS, J. U. MacEWAN and H. H. YATES

## Introduction

**A**N EARLIER paper by MacEwan and Yates<sup>1</sup> describes the corrosion behavior of a variety of welded steel panels. After twelve months alternate immersion in 3 percent NaCl solution all mild steel panels gave some indication of increased attack of the parent metal adjacent to the weld. In contrast, panels of SAE 4130 steel (0.55 percent Mn, 1.0 percent Cr, 0.2 percent Mo, 0.3 percent C) all showed protected zones on each side of the weld bead. The extent of protection varied from the order of  $\frac{1}{2}$  inch for gas welded panels (specimen A of Figure 1) to  $\frac{1}{8}$  inch or less for some electric welded panels (specimen B of Figure 1). Microscopic examination revealed that the protected zone coincided very closely with the region of altered metallurgical structure caused by the temperature cycle of the welding operation. This paper offers a possible explanation for such behavior based on a study of the effect of heat treatment on the galvanic activity of 4130 steel.

## Experimental

The general appearance of 4130 test panels (Figure 1) suggested a definite anode-cathode relationship between normal parent metal and the heat affected material adjacent to the weld. The nature of this relationship was examined by measuring short circuit current between ordinary plate metal and variously heat treated specimens. Although welded test panels used in the previous work were prepared from  $\frac{1}{8}$  inch plate, specimens for the present study were cut from more readily available  $\frac{1}{4}$  inch plate. Chemical analysis and microscopic examination both failed to reveal any significant difference between the two materials.

It had already been established<sup>2</sup> that the zero resistance ammeter circuit proposed by Brown and Mears<sup>3</sup> could be used to establish a qualitative picture of galvanic action at a weld. The same technique was adopted for this investigation. The metals of a given couple were mounted in lucite as shown in Figure 2. Exposed surfaces were  $\frac{1}{2}$  by  $\frac{1}{8}$  inch with a separation of  $\frac{1}{8}$  inch. Electrical contact was made through small mercury pools placed in holes drilled in the lucite mounting. Leads of 20 gauge solid copper wire were held in place with paraffin wax. Following abrasion with polishing paper down to No. 00, the last sequence being parallel to the long dimension of the specimens, the sample mount was attached with paraffin wax to a lucite holder as shown in Figure 2. The surfaces of the couple were wiped carefully with tissue dipped in carbon tetrachloride and connections to the zero resistance ammeter were made. The unit was immediately placed in a beaker of solution and current measurements were begun.

To conform with previous tests<sup>1</sup> a gas mixture of 20 percent O<sub>2</sub> and 80 percent N<sub>2</sub> was saturated with water vapor and bubbled at the rate of 30 cc/min into the solution of 3 percent NaCl in distilled water. Relative positions of bubbler and couple were easily adjusted as indicated in Figure 3 and the entire unit was suspended in a water bath maintained at  $25.0^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$ .

Following an initial rise to a broad maximum the current was found to decrease slowly due to build-up of corrosion products. Values corresponding to the broad maximum were reasonably reproducible and were adopted for this study. Occasional questionable values were found to coincide with the appearance of mechanically deposited rust on the cathodic specimen. This was particu-



J. U. MacEWAN is chairman of the McGill University Department of Metallurgical Engineering and holds the Chair of Birks Professor of Metallurgy. He graduated from Queen's University, Kingston, Ontario, in 1922. He was on the staff of the Montana School of Mines from 1931 until 1936.

D. C. BRIGGS is with the Physical Metallurgy Division of the Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada. He holds the degrees of B.Eng ('56) and M.Eng ('58) from McGill University where, as a post-graduate student, he was engaged in corrosion research. His present work entails investigation of alloy systems of interest in high temperature applications.



H. H. YATES is an associate professor in the McGill University Department of Metallurgical Engineering. He received a PhD in physical chemistry from that university in 1952. A member of CPEQ, CIM and AIIME, he is chairman of the Montreal ASM chapter and vice-chairman of the Canadian Region of NACE.

## Abstract

Increased resistance to corrosion adjacent to welds in SAE 4130 steel is associated with structural changes caused by thermal factors. Separation and analysis of carbides from variously cooled specimens confirm that rapid cooling prevents the normal concentration of chromium in the carbide phase. Galvanic studies show that the resulting material, with a higher than normal chromium content in the ferrite, is definitely more cathodic than slowly cooled material. Microscopic examination also suggests a secondary effect due to the size, and possibly the shape of carbide particles. Transfer of molybdenum to the carbide phase was also noted, but its dependence on cooling rate was rather hazardous. Possible minor effects on galvanic behavior were masked by the overwhelming influence of chromium. 3.7.3

larly prone to happen with weak galvanic couples for which reversals of current were occasionally noted.

Samples were heat treated and identified according to Table 1. All such samples were coupled with a specimen of the metal as received (hot rolled and process annealed—designated AR). Four separate determinations of short circuit current were made with each couple. To minimize possible effects due to movement of solution, two of each four measurements were made with the couple inverted. Although average values of current listed in Table 2 all show a flow from as received metal to heat treated metal, one reading each for samples 1700 FC and 1700 IT were in the reverse direction.

All specimens were examined under the microscope and the structures are listed in the last column in Table 2. Photomicrographs of typical structures are shown in Figure 4 for comparison

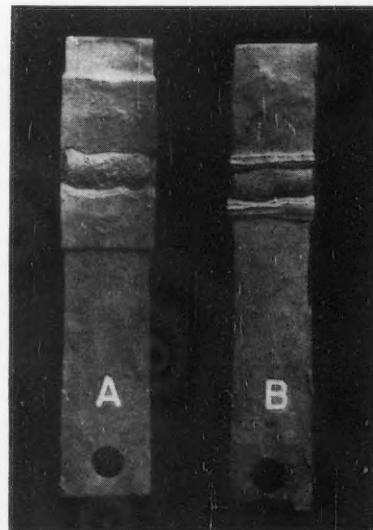


Figure 1—Alternate immersion panels of 4130 steel.  
A—gas welded; B—electric welded.

\*Submitted for publication September 8, 1959.

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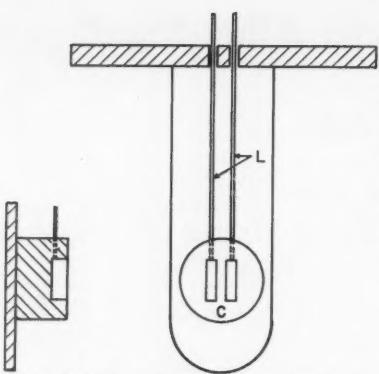


Figure 2—Lucite sample mount and holder. C—mounted couple; L—leads.

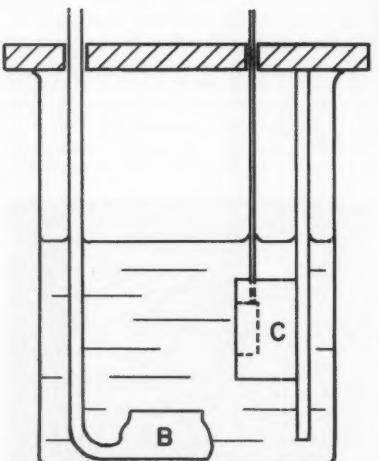
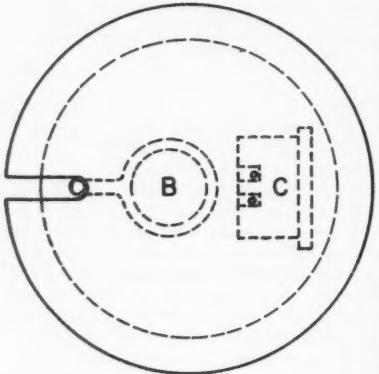


Figure 3—Arrangement of holder and bubbler in beaker. B—bubbler; C—mounted couple.

with actual weld zone structures given in Figure 5.

It is well known that alloying elements do not generally distribute uniformly between the phases of a duplex alloy. An attempt was made, therefore, to establish the effect of temperature cycle on the distribution of chromium and molybdenum between the ferrite and carbide phases in 4130 steel. Samples of carbide were obtained electrolytically from all

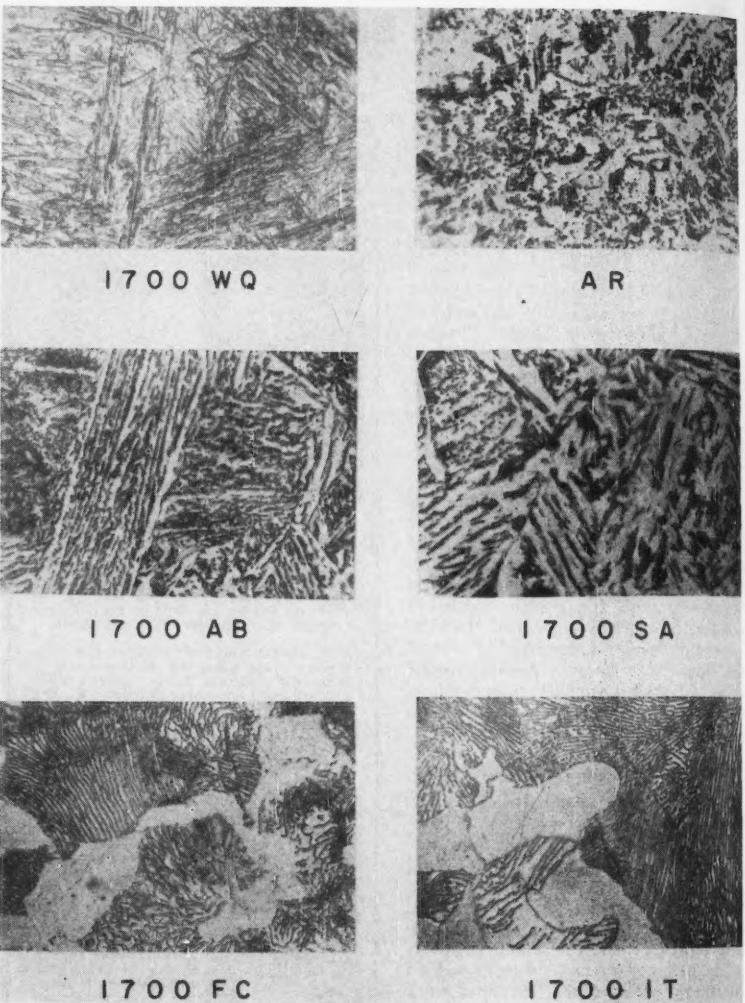


Figure 4—Typical photomicrographs of samples used in short circuit current tests (see Table 1). 1250X.

TABLE 1—Identification and Heat Treatment of Samples.

Austenitizing Temperature, Degrees F.	Method of Cooling	Sample Designation
2050 .....	Air blast	2050 AB
2050 .....	Still air	2050 SA
1700 .....	Water quench	1700 WQ
1700 .....	Air blast	1700 AB
1700 .....	Still air	1700 SA
1700 .....	Furnace cool	1700 FC
1700 .....	Furnace cool to 1300° F., held 3 hr., furnace cool to room temperature	1700 IT
1550 .....	Water quench	1550 WQ

and carbon; results are given in Table 3. Comparison of the structural features of these materials can be made by reference to the photomicrographs in Figure 4 (specimen AR) and Figure 5 (specimen A). The general distribution of carbide is more uniform in the  $\frac{1}{8}$  inch plate but the difference is not great, especially when it is realized that considerable variation was noted within given specimens of each type.

#### Discussion

Short circuit current results in Table 2 and the corrosion pattern of welded 4130 test panels (Figure 1) both suggest a definite trend toward stronger cathodic behavior as the rate of cooling is increased. A number of factors have been considered in seeking an explanation for these results and the following paragraphs offer a reasonably consistent picture based upon the partition of alloying elements between phases and the size and distribution of carbide particles. The oxide film formed during welding<sup>4</sup> may have been strong enough in the case of 4130 steel to withstand subsequent wire

brush treatment and hence to afford some protection to the affected zone. However, this seems to be an unlikely source of protection for prolonged exposure to chloride solution and must certainly be disregarded in the case of current test surfaces which were abraded with polishing paper just prior to use.

#### The Partition of Alloying Elements

The distribution of chromium between carbide and ferrite phases is suggested as the dominant factor governing the formation of protected zones adjacent to welds and also the galvanic behavior of heat treated test pieces. It is well established that chromium in steel tends to concentrate in cementite rather than in ferrite or austenite,<sup>6</sup> and may replace iron in this phase up to 15 percent.<sup>5,7</sup> It is further established<sup>6,8</sup> that cementite is the only carbide formed in the composition range of 4130 steel.

Now when austenite transforms rapidly there is not sufficient time for chromium, with a slow diffusion rate, to migrate to the carbide nuclei; the chromium contents of cementite and ferrite phases are little changed from that of the steel. As the transformation rate is decreased, however, more time is available for diffusion and the chromium content of the cementite rises with attendant depletion of the ferrite. This trend is very evident in the analyses of Table 2 which also suggest that the material as received was cooled relatively slowly through the transformation range. The gradual change from weak cathodic behavior for slow cooled specimens containing low chromium ferrite to strong cathodic behavior for fast cooled specimens containing high chromium ferrite is quite consistent with potential results reported by Uhlig et al.<sup>9</sup>

The possibility of an effect due to partition of molybdenum was also considered, but the random nature of the analytical results for this element in Table 2 belies the existence of any strong influence. It is still conceivable, of course, that a minor contribution has been masked by the overwhelming effect of chromium. A similar study of some chromium free molybdenum steel (possibly 4615) would be of interest in this respect.

#### Size and Distribution of Carbide Particles

Data in Table 2 for samples 1700 FC and 1700 IT appear to dispute the suggested influence of chromium partition. Both samples were slightly cathodic to AR in spite of a lower chromium ferrite. It is true that current was reversed in one of the four measurements for each of these samples and that accuracy is somewhat doubtful for small current values, but it is felt that the cathodic relationship suggested by average values of current is quite valid. The following explanation for such apparently anomalous behavior is based upon the size and distribution of carbide particles.

It has been pointed out by numerous authors that lattice distortion in the vicinity of grain boundaries is associated with increased energy over the normal value within the crystals. This boundary

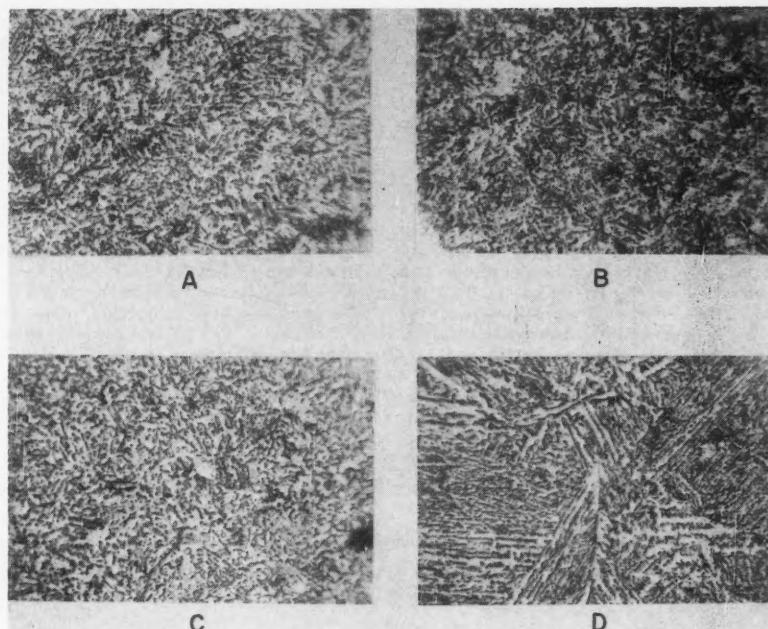


Figure 5—Photomicrographs of welded test panel. A—unaffected parent metal; B—5 mm from weld; C—4 mm from weld; D—2 mm from weld. 1250 X.

TABLE 2—Results of Current Tests, Chemical Analyses and Micro-examination

Sample	Current*, Microamp.	Percent Cr.		Percent Mo	Microstructure
		Carbide	Iron Phase		
1700 WQ.....	48	...	1.05	0.199	Martensite
1550 WQ.....	46	...	1.05	0.199	Martensite
2050 AB.....	16	1.90	1.01**	4.22	.....
1700 AB.....	30	1.77	1.02	2.82	Fine geometric distribution of very fine spheroidal carbide.
2050 SA.....	19	...	....	....	Fine geometric distribution of fine spheroidal carbide.
1700 SA.....	16	1.94	1.01	3.29	Medium geometric distribution of fine spheroidal carbide.
AR.....	...	6.75	0.77	3.26	Random cluster distribution of fine spheroidal carbide.
1700 FC.....	8	7.72	0.71	3.03	Pearlite plus ferrite
1700 IT.....	3	9.88	0.61	2.69	Pearlite plus ferrite

\* In all cases average current flow was from as received metal to heat treated metal.

\*\* For two phase steels

$$\% \text{ Cr (iron phase)} = \frac{\% \text{ Cr (steel)} - \frac{\% \text{ Cr (carbide)} \times \% \text{ carbide (steel)}}{100}}{100 - \% \text{ carbide (steel)}} \times 100$$

TABLE 3—Analyses of SAE 4130 Steel Plate

Material	% Cr	% Mo	% Mn	% C
1/8 in. plate.....	0.99	0.20	0.55	not obtained
1/4 in. plate.....	1.05	0.199	0.58	0.32

energy and its effect on reactivity and corrosion are considered at length in the Electrochemical Society Symposium<sup>10</sup> and also by Evans.<sup>11</sup> It is reasonable to suppose that most of the distortion associated with a ferrite-carbide interface will occur in the softer, weaker ferrite which should, therefore, become more reactive

as the amount of interface increases (i.e. as the carbide particles become smaller). The cathodic behavior of samples 1700 FC and 1700 IT is thus associated with the coarse pearlitic carbide (Figure 4) compared with the fine spheroidal carbide of sample AR—the difference in distortion being more than great enough

to compensate for slight differences in chromium content of the ferrite. There may also be a shape factor leading to lower distortion associated with plate carbide than with spheroidal carbide.

A consideration of the weld sections in Figure 5 suggests that only section "D" was austenitized during the welding and that it cooled at a rate comparable to air blasting. Increased corrosion resistance can here be attributed to chromium distribution. Section "B" and "C" do not appear to have been austenitized, and enhanced corrosion resistance must be attributed to growth of carbide particles. This is obvious in the case of section "C" but somewhat questionable in the case of "B."

In summary it may be stated that as the cooling rate of 4130 steel from austenitic temperatures is increased its cor-

rosion resistance is increased due to retention of greater amounts of chromium in the ferrite. At very slow cooling rates secondary effects associated with the size, and possibly the shape, of carbide particles act in opposition to the chromium effect. Enhanced corrosion resistance of this steel adjacent to welds is attributed to these factors.

It must be emphasized that the change in corrosion resistance is not great and is probably effective only when different samples are in contact. It is very doubtful that a series of specimens, cooled at different rates and subjected separately to the same environment, would show appreciable differences in behavior. However, where intimate contact of variously heated parts is unavoidable, a prediction should be possible concerning the resulting corrosion pattern.

#### Acknowledgment

Financial assistance from the Defence Research Board of Canada is gratefully acknowledged.

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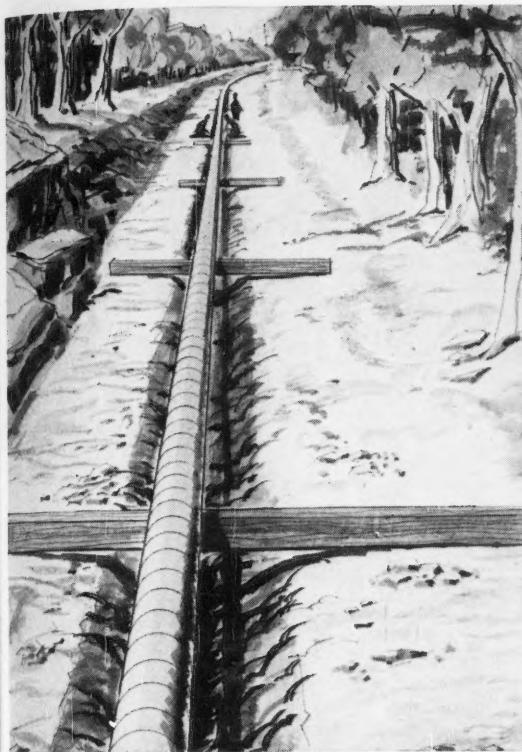
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Durco high silicon cast irons are resistant to sea, brackish and fresh waters as well as all soil environments.

Duriron Anodes have unexcelled corrosion resistance, good electrical properties, and they are easy to install. For real protection, get Duriron. Write for Bulletin DA/6.

#### STANDARD DURIIRON ANODE SIZES

Type Anode	Size	Area Sq. Ft.	Weight (Lbs.)	General Application
B	1" x 60"	1.4	14	Fresh Water
C*	1½" x 60"	2.0	25	Ground Bed
D	2" x 60"	2.6	46	Ground Bed
E	3" x 60"	4.0	115	Salt Water
G	2" x 9"	0.4	5	Ducts
J	3" x 36"	2.5	80	Salt Water
M	2" x 60"	2.8	57	Salt Water

\*Applicable also in fresh water service. Should not be used in ground beds without backfill.

**THE DURIIRON COMPANY, INC.**  
**DAYTON, OHIO**



# What's your "PIPELINE I.Q."?



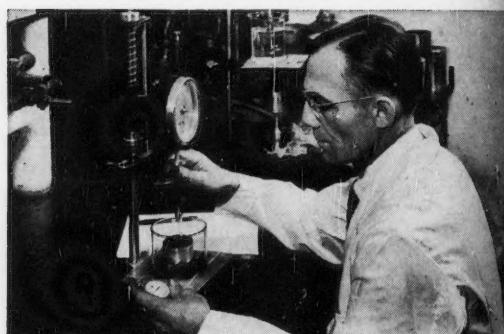
1 During the next ten years, how much money will be invested in new domestic transmission facilities?  
(a) \$3 billion; (b) \$6 billion; (c) \$16 billion.



2 The cost of coal tar pipeline enamel amounts to what percentage of a pipeline's total cost?  
(a) 1½%; (b) 2½%; (c) 4%.



3 This coal tar-protected pipeline, buried in salt water laden soil, was found in perfect condition after:  
(a) 12 years; (b) 17 years; (c) 23 years of service.



4 What coal tar enamel manufacturer produces every drum of its pipeline enamel to written specifications?

## ANSWERS

1 Sixteen billion dollars! This investment is worth the finest protection available.

2 Only one and one-half percent! So why gamble on "economy type" enamels or unproven protective methods when you can have the *proven long-time protection* of coal tar coatings.

3 Twenty-three years! No other popular type of pipeline enamel can begin to match the ability of coal tar enamel to resist moisture absorption.



New Pipeline Guide!  
Just off the press . . . a 16-page booklet which provides the basic facts needed for the selection and most efficient application of pipeline enamel on any pipeline job. Send for your free copy today.

4 Pittsburgh Coke & Chemical Company! It's your guarantee of consistently superior quality and performance. In addition, Pittsburgh Coke backs every drum of Pitt Chem Pipeline Enamel with the services of a full-time staff of field service men who work with your field men in the efficient, economical application of coatings. Write us about your pipeline protection requirements today!



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